PB82-214222

Determination of Pesticides and PCB's in Industrial and Municipal Wastewaters

Southwest Research Inst.
San Antonio, TX

Prepared for

Environmental Monitoring and Support Lab.
Cincinnati, OH

Apr 82

U.S. Department of Commerce
National Technical Information Service
NTIS

PB82-214222

EPA-600/4-82-023
April 1982

DETERMINATION OF PESTICIDES AND PCB's IN INDUSTRIAL AND MUNICIPAL WASTEWATERS

by

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Contract No. 68-03-2606

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SPRINGFIELD, VA 22161

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/4-82-023	2. ORD Report	3. RECIPIENT'S ACCESSION NO. PB82-214222
4. TITLE AND SUBTITLE Determination of Pesticides and PCBs in Industrial and Municipal Wastewaters		5. REPORT DATE March 1982
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) John D. Millar, Richard E. Thomas, Donald E. Johnson		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Southwest Research Institute 8500 Culebra Road San Antonio, Texas 78284		10. PROGRAM ELEMENT NO. IBD612
		11. CONTRACT/GRANT NO. 68-03-2606
12. SPONSORING AGENCY NAME AND ADDRESS Environmental Monitoring and Support Laboratory Office of Research and Development U.S. Environmental Protection Agency Cincinnati, Ohio 45268		13. TYPE OF REPORT AND PERIOD COVERED Final
		14. SPONSORING AGENCY CODE EPA/600/06

15. SUPPLEMENTARY NOTES

16. ABSTRACT

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The method is satisfactory for the analysis of clean waters and wastewaters having a relatively low background of interferences. However, it does not work well against medium to high levels of background interferences produced by substances that are electron capture sensitive, especially halogenated ones. Use of the Hall detector is indicated when nonhalogenated electron capture sensitive interferences are a problem, even though some loss in sensitivity will occur. When halogenated interferences are overwhelming, altered gas chromatography conditions and columns, such as temperature programming and columns which produce better resolution than the ones studied in this work, will be required.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group

18. DISTRIBUTION STATEMENT Release to public	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES
	20. SECURITY CLASS (This page) Unclassified	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

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FOREWORD

Environmental measurements are required to determine the quality of ambient waters and the character of waste effluents. The Environmental Monitoring and Support Laboratory - Cincinnati conducts research to:

- o Develop and evaluate techniques to measure the presence and concentration of physical, chemical, and radiological pollutants in water, wastewater, bottom sediments, and solid waste.
- o Investigate methods for the concentration, recovery, and identificiation of viruses, bacteria, and other microbiological organisms in water. Conduct studies to determine the responses of aquatic organisms to water quality.
- o Conduct an Agency-wide quality assurance program to assure standardization and quality control of systems for monitoring water and wastewater.

Under provisions of the Clean Water Act, the Environmental Protection Agency is required to promulgate guidelines establishing test procedures for the analysis of pollutants. The Clean Water Act Amendments of 1977 emphasize the control of toxic pollutants and declare the 65 priority pollutants and classes of pollutants to be toxic under Section 307(a) of the Act. This report is one of a series that investigates the analytical behavior of selected priority pollutants and suggests a suitable test procedure for their measurement.

Robert L. Booth

Robert L. Booth, Acting Director
Environmental Monitoring and Support
Laboratory - Cincinnati

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PAGE NUMBER
EPA-287 (Cin.)
(4-76)

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The method is satisfactory for the analysis of clean waters and wastewaters having a relatively low background of interferences. However, it does not work well against medium to high levels of background interferences produced by substances that are electron capture sensitive, especially halogenated ones. Use of the Hall detector is indicated when nonhalogenated electron capture sensitive interferences are a problem, even though some loss in sensitivity will occur. When halogenated interferences are overwhelming, altered gas chromatography conditions and columns, such as temperature programming and columns which produce better resolution than the ones studied in this work, will be required.

This report was submitted in partial fulfillment of Contract 68-03-2606 by Southwest Research Institute under the sponsorship of the U.S. Environmental Protection Agency. This report covers a period from September, 1977 to February, 1979, when work was completed on the developmental phase of the contract.

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ACKNOWLEDGMENTS

The following personnel of Southwest Research Institute gave especially valuable assistance in the laboratory during this program: H. J. Schattenberg, C. P. Amox, R. C. Robledo, and H. J. Harding. O. Saenz, Jr., also of Southwest Research Institute, is gratefully recognized for procuring two wastewater samples.

Three other wastewater samples were procured under the auspices of J. E. Longbottom, Project Officer, whose assistance in this and other matters was very helpful.

SECTION 1

INTRODUCTION

This project was part of an overall program of the Environmental Protection Agency (EPA) to establish test procedures for the analysis of organic toxic substances in wastewaters. These toxic compounds have been separated into 12 categories as follows:

Category 1: Phthalate Esters
Category 2: Haloethers
Category 3: Chlorinated Hydrocarbons
Category 4: Nitrobenzenes and Isophorone
Category 5: Nitrosamines
Category 6: Dioxin
Category 7: Benzidines
Category 8: Phenols
Category 9: Polynuclear Aromatics
Category 10: Pesticides and PCB's
Category 11: Purgeables
Category 12: Acrolein, Acrylonitrile, Dichlorodifluoromethane

This report is on Category 10: Pesticides and PCB's.
The substances studied in this effort are:

1. α-BHC
2. β-BHC
3. δ-BHC
4. Heptachlor epoxide
5. DDE (p,p')
6. DDD (p,p')
7. DDT (p,p')
8. Endosulfan sulfate
9. γ-BHC
10. Heptachlor
11. Aldrin
12. Endosulfan I
13. Dieldrin
14. Endrin
15. Endosulfan II
16. Endrin aldehyde
17. Chlordane
18. Toxaphene
19. PCB-1016
20. PCB-1221
21. PCB-1232
22. PCB-1242
23. PCB-1248
24. PCB-1254
25. PCB-1260

The study was conducted in two phases. In Phase I, work was conducted with clean water and was intended to provide information that would give direction to Phase II work, conducted on actual wastewaters, and serve as a comparison base for the information developed.

The ultimate objective was to develop a method, having a maximum of applicability, that could be published in the Federal Register and carried out in most analytical laboratories.

Other objectives were to develop accuracy and precision information for the various steps in the method, to develop procedure variations as required by a particular wastewater, and to maximize the use of common sample treatment steps for all of the categories of organic toxic substances listed above.

SECTION 2

CONCLUSIONS

The 25 substances can be gas chromatographed very well. Sensitivity quantities (producing a response 10X noise level) for the electron capture detector range from 1 to 380 picograms. Sensitivity quantities for the Hall electrolytic conductivity detector are roughly 10 to 100 times greater than for the electron capture detector. The 16 single-compound pesticides can be fully resolved if chromatographed in two groups of 8 each. The PCB's are chromatographed best individually, but several combinations of them can be chromatographed adequately for some analytical purposes.

Extraction of the pesticides from clean water at parts per trillion concentrations when the pH was 2, 7, or 10 was generally 80 percent or better using 15 percent DCM in hexane or pure DCM as the extracting solvent, except for heptachlor and aldrin which were generally 50 to 75 percent recovered. Occasionally, one of the solvents at a particular pH gave a more efficient extraction than did the other solvent-pH combinations, although the improvements were not great enough to demand the particular combination be adhered to exclusively. Under the same conditions of extraction, PCB-1242, PCB-1248, PCB-1254, and PCB-1260, at low parts per billion levels, were extracted with 90 percent or better recoveries. PCB-1016, PCB-1221, and PCB-1232, at similar levels, were extracted with adequate efficiency but the recoveries recorded were occasionally as low as 60 percent.

At the concentrations mentioned above, some of the conditions of the preservation study produced pronounced effects on the substances. Deleterious effects were found to occur with about half of the 25 substances at some of the 12 combinations of pH, temperature, and residual chlorine levels studied. However, 23 of the substances were stored in the dark for 7 days at 4°C, under neutral or acidic pH, and in the presence and absence of 2 parts per million residual chlorine with the only losses suffered being of a tolerable magnitude. Aldrin sustained a 75 to 90 percent loss at acid or neutral pH's in the presence of chlorine. Endosulfan sulfate losses were 90 percent or more under all conditions of storage at 310 parts per trillion concentration in clean water. In the storage and recovery tests involving wastewater, endosulfan sulfate was dosed at the 15 parts per billion concentration and no large losses were observed.

Endosulfan sulfate was found to be unstable in solution in acetone in the course of this program. It was found to be stable in hexane, benzene, toluene, and methyl alcohol.

At the concentrations used in the work with clean water, the principal problems were contaminations introduced from reagents, glassware, and unknown sources during procedural operations that interfered with the quantitative determinations at times. Even the most stringent precautions did not always result in acceptably low and uniform blanks.

Recoveries during column chromatography of substances applied to fully activated Florisil PR or alumina mixed with 10 percent by weight water were near 100 percent. Elution patterns of the substances from the Florisil were more desirable than from the alumina. In application studies with wastewaters, the Florisil was slightly superior to the alumina in cleanup of extracts.

Only a few of the 25 substances were found in the five wastewaters analyzed. Endrin, which was tentatively identified in Wastewater 5, was the only one of the substances present above the 10 parts per billion level. Its concentration was estimated at 32 parts per billion.

Recovery of the substances dosed into the five wastewaters at the 1 to 200 parts per billion level was satisfactory to very good with respect to accuracy and precision for Wastewaters 1, 3 and 4. Gas chromatographic analysis to determine the recoveries from Wastewaters 2 and 5 could not be performed in some instances because of interferences that were not sufficiently removed by cleanup columns, agitation with mercury, alkaline hydrolysis, and/or extraction with sulfuric acid. It is very probable that Wastewater 2 would have been analyzed with greater success with the Hall detector than with the electron capture detector. Since Wastewater 5 was from a pesticide plant, the limited analytical success with this wastewater is indicative of the limitations imposed on the method by high concentrations of chlorinated substances that are not among the 25 substances of interest in this program but that have extraction and chromatography characteristics similar to them. Therefore, neither the Hall detector nor the electron capture detector is completely satisfactory in this situation. Better gas chromatography resolution or separation of such interferences before gas chromatography is required.

In summary, a method has been developed that is satisfactory when used with wastewaters having a relatively low background of interferences. However, it does not work well against medium to high levels of background interferences produced by substances that are electron capture sensitive, especially halogenated ones.

SECTION 3

RECOMMENDATIONS

The primary gas chromatography column used in this work (1.5 percent SP 2250 + 1.95 percent SP 2401 on 100/120 mesh Supelcoport) is preferred on a general use basis over 3 percent OV-1 on the same solid support. However, in limited situations, the OV-1 column, or possibly columns with other liquid phases, such as OV-17, might be as good as or better than the primary column.

The Hall electrolytic conductivity detector is more discriminating than the electron capture detector, and it should be resorted to as the primary detector, or as an auxiliary detector, when nonhalogenated electron capture sensitive materials present interference problems.

No clear advantage has been noted for either solvent studied in this work. In the interest of commonality, either solvent may be chosen without an intolerable loss of efficiency occurring during sample extraction. The pH of the water at the time of extraction is not of substantial concern. However, if the substance being sought is designated beforehand, a slight advantage may be available in some instances by consulting the statistical analysis data for combined pH-solvent effects on the extraction of that particular substance.

Storage of samples before extraction should be at neutral to acidic pH and at 4°C. Storage periods of up to 7 days' duration do not produce significant decreases in concentrations, except for endosulfan sulfate in concentrations of parts per trillion. For determinations at these levels, samples should be extracted immediately after sampling. If aldrin is to be determined, residual chlorine, if present, should be eliminated before storage of samples.

Endosulfan sulfate should not be kept in solution in acetone. Hexane, benzene, toluene and methyl alcohol are satisfactory solvents for this compound.

Fully activated Florisil PR is preferred over alumina with 10 percent by weight water as the cleanup adsorbent for extracts from samples. No completely satisfactory means of removing sulfur interferences is known. Agitation with mercury has some advantages over other methods.

Great care should be exercised in preparing the reagents and glassware used in the analyses. This is especially so if the substances being analyzed are in the parts per trillion concentrations in the samples. More details on these matters are given in the text of this report. Glassware and reagent blanks should be run from time to time to establish the levels of contamination from these sources.

Quantitation may be obtained through computer integration of peak areas or by peak height measurements. Peak height measurements have often been found to produce more reliable data than computer integration, especially where substances giving multiple peaks are concerned. In the case of the multipeak substances, all of the major peaks free from gross interferences should be used independently to determine the quantity of substance present.

Owing to the complexities of the method, only chemists experienced with the method, or persons under their close supervision, should conduct analyses for the 25 substances.

It should be realized that the applicability of the method can be severely limited by interferences that cannot be removed by any means known at this time. Additional work to improve cleanup of extracts is indicated. Some possibilities are: two cleanup columns, such as Florisil plus silica gel or alumina plus cocoanut charcoal; improved chemical treatment to destroy the interferences; gel permeation chromatography, even though this is best suited to removing lipids, which were not a problem on this program. In conjunction with work to improve cleanup of extracts, alteration of gas chromatography conditions and columns as possible means to improve resolution should be explored, especially longer packed columns and capillary columns 10 to 20 meters in length. Cleanup requirements may be reduced substantially by improving resolution.

SECTION 4

PHASE I

OBJECTIVES

The objectives of Phase I were limited to:

1. The demonstration of the gas chromatography (GC) of all 25 substances on two analytical columns, employing an electron capture detector and a halogen specific detector. The sensitivity of each detector was to be determined for each substance on at least one column.
2. The determination of extraction efficiency of two solvents for obtaining the substances from clean water at three pH levels.
3. The determination of the effects on the substances of storage for seven days at two temperatures, three pH levels, and with or without free chlorine in the water.

PROGRAM DIRECTION

Before experimental work began, representatives of SwRI met in Cincinnati with the project officer and others of concern in the EPA Cincinnati laboratories to discuss and decide on certain specific aspects of the activities in Phase I. In addition to the decisions reached on GC columns, extraction solvents, and other experimental details which will be given in subsequent sections of this report, guidelines were developed as follows:

- Unusual, exotic, or overly expensive items are not desirable. Simplicity is to be preferred wherever the option exists. For example, mass spectrometry and WCOT or SCOT columns are ruled out.
- Use of chemical treatment to produce a derivative which cannot be unequivocally related to the parent substance is not acceptable.
- PCB's must be identified individually by number, not as "total PCB's", using as many peaks as practicable.
- Quantitation by electronic integration of peak area or by peak height measurement is acceptable.

EXPERIMENTAL

Gas Chromatography

All 25 substances were chromatographed on two columns. The primary column (Column 1) is 1.8m × 6.35mm glass tubing packed with 1.5 percent SP-2250 plus 1.95 percent SP-2401 on 100/120 mesh Supelcoport. The secondary column (Column 2) has 3 percent OV-1 as its liquid phase but is otherwise the same as Column 1.

Using Column 1, it is possible to achieve baseline resolution for the sixteen pesticides if they are separated into two groups as follows:

Group I	*Group II*
α-BHC	γ-BHC
β-BHC	Heptachlor
δ-BHC	Aldrin
Heptachlor epoxide	Endosulfan I
DDE	Dieldrin
DDD	Endrin
DDT	Endosulfan II
Endosulfan sulfate	Endrin aldehyde

The other two pesticides, chlordane and toxaphene, and the seven PCB's were each treated as separate groups, giving a total of 11 groups. All of the experiments in the project were run using these 11 groupings. However, it is possible to chromatograph several combinations of the PCB's suitably for analytical determinations. For example: 1221 + 1254, 1016 + 1260, 1242 + 1254, 1232 + 1260, 1221 + 1248, and 1221 + 1016 + 1260.

Column 2, at the operating conditions given below, does not adequately separate peaks produced by α-BHC, β-BHC, and δ-BHC or by endosulfan II and endrin. Therefore, the compounds in Groups I and II, above, must be placed in 3 or more groups for chromatography, or else more than one oven temperature must be used with Column 2. However, 24 of the 25 substances may be chromatographed satisfactorily on an individual basis or in groups, within the restrictions mentioned. Column 2 has a lower bleed level than column 1. Consequently, it exhibits a lower noise level, a feature which can be advantageous at times.

The gas chromatograph used was a Hewlett-Packard 5710A GC, equipped with a Ni^{63} electron capture detector. Operating conditions were usually as follows:

Oven temperature	200°C
Detector temperature	300°C
Carrier gas (5 percent methane in argon) flow rate	60mL/min

Other conditions may be used satisfactorily. For example, in this work, it was sometimes advantageous to chromatograph several of the PCB's at a lower temperature.

Figures A1 through A11 are chromatograms, obtained with Column 1, of all 25 substances in the 11 groups studied. For several of the PCB's, there are chromatograms at two temperatures: 200°C and 160°C. The numbers on chromatograms of substances producing multiple peaks identify the peaks used to determine the recovery of those substances in these studies. Toxaphene peaks 7 and 8 were not used in Phase I but were used in Phase II as a result of improved programming procedures with the computer-based peak integrator.

Figures A12 through A30 are individual chromatograms obtained for all 25 substances using Column 2 at 200°C.

Retention times relative to aldrin and absolute retention times on Column 1, the preferred column on a general use basis, are given below in Table 1.

TABLE 1. RETENTION TIMES
(Column 1: 200°C; 60 mL/min)

Substance	*Min*	*RRT*
α-BHC	1.61	0.55
γ-BHC	2.02	0.69
β-BHC	2.29	0.78
Heptachlor	2.46	0.84
δ-BHC	2.64	0.90
Aldrin	2.93	1.00
Heptachlor epoxide	4.37	1.49
Endosulfan I	5.48	1.87
DDE (p,p')	6.24	2.13
Dieldrin	6.65	2.27
Endrin	8.06	2.75
DDD (p,p')	9.52	3.25
Endosulfan II	9.87	3.37
DDT (p,p')	11.43	3.90
Endrin aldehyde	12.86	4.39
Endosulfan sulfate	15.59	5.32

	Peak Number										
Substance	1	2	2	4	5	6	7	8	9	10	11
	Min.										
Chlordane	2.26	2.46	3.34	4.83	5.22	9.29					
Toxaphene	6.59	8.61	9.26	9.87	10.84	11.95	15.21	20.51			
PCB-1016	1.49	1.88	2.40	2.64	2.81	3.02	3.52	3.75			
PCB-1221	0.91	1.11	1.23	1.32	1.49	1.90	2.40				
PCB-1232	0.91	1.35	1.49	1.90	2.40	2.64	2.81	3.02	4.42	5.19	
PCB-1242	1.49	1.88	2.40	2.64	2.81	3.02	3.52	4.42	5.19		
PCB-1248	1.88	2.40	2.99	3.52	3.75	4.42	5.19				
PCB-1254	4.42	5.13	6.80	7.79	8.76	9.90	11.19				
PCB-1260	4.54	5.04	6.97	7.71	8.79	9.87	11.19	12.01	14.01	17.48	22.65
	RRT										
Chlordane	0.77	0.84	1.14	1.65	1.78	3.17					
Toxaphene	2.25	2.94	3.16	3.37	3.70	4.08	5.19	7.00			
PCB-1016	0.51	0.64	0.82	0.90	0.96	1.03	1.20	1.28			
PCB-1221	0.31	0.38	0.42	0.45	0.51	0.65	0.82				
PCB-1232	0.31	0.46	0.51	0.65	0.82	0.90	0.96	1.03	1.51	1.77	
PCB-1242	0.51	0.64	0.82	0.90	0.96	1.03	1.20	1.51	1.77		
PCB-1248	0.64	0.82	1.02	1.20	1.28	1.51	1.77				
PCB-1254	1.51	1.75	2.32	2.66	2.99	3.38	3.82				
PCB-1260	1.55	1.72	2.38	2.63	3.00	3.37	3.82	4.10	4.78	5.97	7.73

Sensitivity values obtained for the substances using electron capture detection are given in Table 2. In the case of substances producing multiple peaks, values were based on measurement of the tallest peak or one of the two tallest peaks. Column 2 gave the higher sensitivity values, probably because of its shorter elution times, its greater stability and consequent lower noise level.

TABLE 2. SENSITIVITY VALUES-ELECTRON CAPTURE DETECTOR

	Sensitivity Level (10X noise level),pg	
Substance	*Column 1*	*Column 2*
α-BHC	2	0.8
β-BHC	4	2
γ-BHC	2	1
δ-BHC	4	2
Heptachlor	2	1
Aldrin	3	2
Heptachlor epoxide	4	2
Endosulfan I	5	2
DDE (p,p')	6	2.5
Dieldrin	6	2.5
Endosulfan II	10	4
Endrin	9	12.5
DDD (p,p')	12	7
DDT (p,p')	16	8
Endrin aldehyde	23	5
Endosulfan sulfate	31	11
Chlordane	38	17
Toxaphene	380	111
PCB-1016	43	14
PCB-1221	100	50
PCB-1232	100	33
PCB-1242	50	20
PCB-1248	75	33
PCB-1254	75	33
PCB-1260	150	50

Sensitivity determinations were also made using Column 2 installed in an MT 220 gas chromatograph equipped with a Tracor 310 Hall Electrolytic Conductivity Detector. Operating conditions were as follows:

GC oven temperature	180 or 200°C
Furnace temperature	840°C
Empty quartz tube in furnace	
Electrolyte fluid	absolute ethyl alcohol
Carrier gas (He) flow	35 mL/min
Hydrogen gas flow to furnace	10 mL/min
Transfer line temperature	225°C
Chlorine mode resin cartridge in detector	
Attenuation	1 X
Vent time	1.5 to 2 min

It was necessary to chromatograph some of the more volatile substances at 180°C instead of 200°C to prevent elution of some of the major peaks during the vent period. The sensitivity values, which are based on the tallest peak in the instance of multiple peak substances, are given in Table 3.

TABLE 3. SENSITIVITY VALUES-TRACOR 310 HALL DETECTOR

Substances	*Sensitivity Level (10X noise level), ng*
α-BHC*	0.6
β-BHC*	0.7
γ-BHC*	0.7
δ-BHC*	1.1
Heptachlor*	0.5
Aldrin	0.6
Heptachlor epoxide	1.3
Endosulfan I	0.6
DDE (p,p')	6.3
Dieldrin	0.8
Endosulfan II	0.9
Endrin	3.2
DDD (p,p')	3.6
Endrin aldehyde	1.3
DDT (p,p')	3.2
Endosulfan sulfate	3.8
Chlordane	3.2
Toxaphene	6.1
PCB-1260	15.3
PCB-1254	10.1
PCB-1248	6.0
PCB-1242*	9.8
PCB-1232*	8.7
PCB-1221*	4.5
PCB-1016*	5.5

*GC oven at 180°C; all others at 200°C.

A representative of the manufacturer of the Hall Detector, Tracor Instruments, states that Model 700A has the capability of being about ten times more sensitive than the Model 310 used in this work.

Extraction Study

Procedures and Results--

The extraction study was initiated to determine the recoveries of the 25 substances of interest from clean water at pH 2, 7 and 10 using 15 percent dichloromethane (DCM) in hexane and 100 percent DCM as the extracting solvents.

The water used in the extraction study is a naturally buffered well water obtained from the SwRI supply line prior to chlorination. Samples of this water were collected and transported to the laboratory in empty solvent bottles which had previously contained Burdick and Jackson solvents. The pH of the untreated water is close to 8. Adjustment to the required pH's was accomplished by adding strong acid or base as follows:

pH 2 - 2.5mL 18N H_2SO_4
pH 7 - 0.1mL 18N H_2SO_4
pH 10 - 0.2mL 10N NaOH

The water was found to be very low in electron capture sensitive materials as determined through comparison of an extract of the water with a glassware blank (see Figures A31 and A32). Chromatograms in these instances were very similar, strongly indicating that the water could not be the source of any large interferences.

The water was dosed with acetone solutions of the 25 substances, each solution containing 100 times the sensitivity amount per microliter, as determined earlier for each substance. By adding 100μL of the appropriate acetone solution to a liter of water, dosed water samples for extraction contained the quantities of the substances shown in Table 4.

TABLE 4. QUANTITIES ADDED TO WATER SAMPLES

Substance	*Quantity added to 1 liter, ng*
α-BHC	20
β-BHC	40
γ-BHC	20
δ-BHC	40
Heptachlor	20
Aldrin	30
Heptachlor epoxide	40
Endosulfan I	50
DDE (p,p')	60
Dieldrin	60
Endosulfan II	100
Endrin	90
DDD (p,p')	120
DDT (p,p')	160
Endrin aldehyde	230
Endosulfan sulfate	310
Chlordane	380
Toxaphene	3800
PCB-1016	1290*
PCB-1221	3000*
PCB-1232	3000*
PCB-1242	1500*
PCB-1248	2250*
PCB-1254	750
PCB-1260	1500

* These quantities are 3X the amount calculated by the procedure given in the text of this report. The increase was necessary to avoid GC operation at lower than 8X attenuation when using an oven temperature of 160°C.

The one-liter samples of water were dosed while in one-liter Erlenmeyer flasks then poured into two-liter separatory funnels for extraction in accordance with the procedure found in the Federal Register[1], using either 15 percent DCM in hexane or 100 percent DCM. Three dosed samples were extracted at pH 2 and 10 with each solvent. At pH 7, four dosed samples were extracted with each solvent. Thus, 20 samples were extracted for each group. The final volume of the extract concentrate was always 10mL and 10μL of the concentrate was always injected into the GC. At the time dosing of the samples occurred, 100μL of the dosing solution was added to 10mL of hexane which then served as the standard against which all recoveries were measured.

The Federal Register procedure calls for the separated extracts to be drained through three to four inches of anhydrous sodium sulfate into a Kuderna-Danish flask. Frequent plugging of the tube containing the sodium sulfate was encountered. Usually unplugging could be achieved by applying a slight pressure to the top of the tube by means of a 2-ounce rubber bulb. It was discovered that the column of sodium sulfate could be decreased in height to three to four centimeters without any obvious effect on the analytical results and with a more rapid delivery of extract into the Kuderna-Danish flask. In the course of unplugging the sodium sulfate tube, a small amount of water was sometimes forced into the Kuderna-Danish flask. This situation produces no harmful effects unless the quantity is so great as to cause an intolerable volume error in determining the final 10mL volume or unless the water phase should be accidentally injected into the GC. The water may be removed by adding sodium sulfate to the lower tube of the Kuderna-Danish flask before transferring its contents to a 10mL volumetric flask.

When the extracting solvent is 100 percent DCM, it is necessary to remove essentially all of the DCM prior to analysis. This was done by taking the extract to a volume of 10 to 15mL, adding 75 to 100mL of hexane and then reconcentrating to the final volume. The DCM extracts usually produced a wider "solvent" peak than those produced by 15 percent DCM in hexane. This peak broadening was not reduced when the amount of hexane added before reconcentration was increased in amounts up to 200mL. The peak broadening incurred is not a serious problem, however.

Occasionally, an emulsion resulted during the extraction process. This occurred usually with the 15 percent DCM in hexane solvent and at pH 2 and 7. After draining off most of the water phase, the emulsion could be broken almost always by shaking or swirling the separatory funnel. When emulsification occurred, the solvent phase was usually not as well separated from the water phase as when emulsification did not occur, resulting in a higher rate of plugging of the sodium sulfate tube.

The most serious problem encountered was the inability to obtain chromatograms of blanks for water, glassware and sodium sulfate which were devoid of interfering peaks. As the study progressed, the problem became less troublesome, although never completely solved, and occasionally work had to be repeated. The pattern of interference was usually, but not always, the same from extract to extract with variation in magnitudes of the peaks. The most frequently experienced pattern showed substantial peaks at about 3.3, 14 and 19.5 minutes with other smaller peaks present. Figures A33 and A34 show examples of this pattern of interference and its variation in replicate water blanks. Whether glassware was cleaned and heated to 275°C overnight before use or was heated for several hours at 450°C before use seemed to have little effect. Early in the study, the sodium sulfate was heated overnight at 450-500°C before use. Some improvement resulted when this practice was supplanted by Soxhlet extraction of the sodium sulfate with DCM for two days[2]. All glassware was solvent-rinsed with acetone and hexane after use and then, unless immediately reused, was rinsed again with hexane just before being put into service. The sodium sulfate, the holding tube and the glass wool plug were all rinsed with DCM and hexane immediately before use. Despite the problem with blanks, it is believed that the data acquired permit reliable conclusions to be drawn with respect to the objectives of this study.

Percent recovery data were obtained by both computer integrated peak areas and by peak height measurement. In the case of chlordane, toxaphene, and the PCB's, each of the peaks being followed in the analyses was used independently to indicate the total amount of substance present. Peak height measurement was relied upon whenever there was reason to be suspicious of the computer printout values. Computer readings seemed to be especially unreliable from PCB's 1016, 1221, 1232, 1242, and 1248. Repeated attempts were made to set the program parameters for satisfactory readings for these groups without much success. The computer system used was the Hewlett-Packard 3354 Data System.

The pesticides, PCB-1254, and PCB-1260 were gas chromatographed at 200°C and the remaining PCB's were done at 160°C. The lower temperature gave better resolution of peaks and in some instances reduced the number of interferences being noticed in the first four minutes of a run.

The data acquired in the extraction study are presented in Tables A1 through A11.

Data Analysis--

The results of the extraction study were submitted to statistical analysis to determine which of the factors affected the recovery efficiency of the method. The statistical model for these data is a two-way analysis of variance (ANOVA) defined by:

$$Y_{ijk} = \mu + \tau_i + \varrho_j + \tau\varrho_{ij} + \varepsilon_{ijk}$$

where

μ = overall mean

τ_i = effect of solvent system used, i =n 1,2

ϱ_j = effect of pH level, j = 1,2,3

$\tau\varrho_{ij}$ = interactive effect between solvent and pH level, and

ε_{ijk} = random error associated with observation Y_{ijk}.

The factors are considered fixed effects, and the five-percent significance level is used on all tests.

The interaction between solvent and pH, if significant, implies that the combination of a particular pH and a particular solvent performed better (worse) than could be predicted by using the results from the two factors taken separately. For example, if a difference of seven percent is observed between solvents, and six percent between pH's, then using the best combination would result in a predicted difference of thirteen percent, the sum of the two differences. If the observed difference were to be 20 percent, then the interaction term in the ANOVA would be significant. This merely implies that the best estimate of the expected recovery should be obtained from the results on that particular combination, not from the pH or solvent means themselves. Interaction is used in terms of a data analytical rather than physical approach.

A factor or interaction is considered to be significant if an F-ratio calculated as part of the ANOVA procedure exceeds the corresponding tabled value at the five-percent significance level. This implies that there was more variability among the levels of a factor, e.g. among the three pH's, than there was among replicate analyses under identical conditions. In some cases, however, the precision of the individual analyses allows differences to be termed significant that are too small to be of any practical importance. This should be considered in evaluating the results contained in this section.

The data from the sixteen single component pesticides were used individually in the ANOVA to determine the recoveries on each pesticide compound distinctly. For the remaining nine materials, an alternative approach was used. Since these consisted of a mixture of closely related compounds of varying degrees of chlorination, it was deemed appropriate to combine the results of all numbered peaks in the chromatogram. An objective function of the form

$$Z_i = \Sigma \left| R_i - 100 \right|$$

was used, where R_i is the percent recovery for peak i. Summing these differences over the peaks in the chromatogram yielded a single value for a given set of conditions (i.e., pH, solvent, replicate), and these results were used in the ANOVA model for the multicomponent materials.

The results for the single-compound pesticides indicate that for 11 of the 16 compounds there was no significant difference in extraction efficiency between the two solvent systems. In the other five, β-BHC, DDD, DDT, endrin, and endosulfan II, 15-percent DCM in hexane gave a higher percent recovery, on average, than did the 100-percent DCM four times, with only endrin exhibiting better recovery with 100-percent DCM. The observed difference between the recoveries in the two solvent systems was no more than nine percent for any of the significant terms, with four or five percent more common.

The pH of the water was a significant factor in the ANOVA for seven of the 16 single-compound pesticides, and there was a lack of consistency in the results obtained. A pH of 2 to 7 was favored on three of the compounds, DDD, DDT, and endosulfan II, pH 10 favored for heptachlor and endrin, and pH's 2 and 10 equivalent and better than pH 7 for endrin aldehyde. The interaction terms were significant on DDT, where the DCM-pH 10 combination was low, on heptachlor epoxide where both solvent systems were low at pH 10, and for γ-BHC, where considerable variation was observed.

For the single-compound pesticides, then, there is a slight tendency for better extraction efficiency with 15 percent DCM in hexane as the extraction solvent. The selection of a recommended pH was guided by the performance of heptachlor and aldrin. The recovery for these two pesticides was higher by 10 percent or more at pH 10 than at either pH 2 or 7, and were still lower than desirable. The specification of a basic pH, however, raises the minimum recovery for any of the compounds to 66 percent.

For the remaining materials, a composite result was obtained based upon the objective function described above. For chlordane, three numbered peaks were used and the ANOVA on these results gave no significant terms. As a result, the conclusion is that either solvent system may be used with no difference noted in the results, and no adjustment of the pH is necessary.

The average deviation from 100 percent for these data was 3.2, indicating that the average percent recovery based on individual peaks ranged between 97 and 103 for chlordane.

Significant differences were noted in the ANOVA's on the toxaphene results. Both the solvent term and the solvent-pH interaction were significant. Fifteen-percent DCM in hexane was the preferred solvent system, with the interaction term indicating the best results were obtained at pH 2. However, the mean differences for the other pH's were indistinguishable from the acidic results when this solvent system was used. As a result, no adjustment of the pH of the water is indicated by these data to be required.

The average deviation from 100 percent for these data was 6.0, which indicates an expected range of recoveries from 94 to 106 percent. As can be seen from the data summary table, values greater and less than 100 percent were obtained for the peaks studied.

For PCB-1254, the ANOVA yielded no significant terms. There was a slight tendency for results at pH 7 to be closer to 100 percent recovery, but the significance level of the F-ratio was approximately six to seven percent and did not meet the stated criterion. As a result, either solvent system may be effectively used in extracting the water for this PCB, and no adjustment of the pH is necessary prior to extraction.

The average deviation from 100 percent for the seven peaks under study was 4.2 percent, giving an expected range of 96 to 104 percent recovery of the compound.

The results of the ANOVA on PCB-1016 yielded two significant terms, the pH of the water and the solvent-pH interaction. Overall, adjustment to pH 7 prior to extraction produced better results, while the recoveries at pH's 2 and 10 were equivalent to each other. For the six peaks studied, the average recovery was closer to 100 percent recovery by approximately 6 percent when the water was at a neutral condition.

The pH-solvent interaction indicated that the best results were obtained when 15 percent DCM in hexane was used at pH 7. This result was considerably closer to 100 percent than the results for 100 percent DCM, but due to the variability in the data, the DCM results at all pH's were statistically indistinguishable from the 15 percent DCM-pH 7 combination. If the 15 percent DCM in hexane is used as the solvent, the adjustment of the pH is necessary. If 100 percent DCM were used, however, no adjustment would be required.

Average deviations from 100 percent recovery were 6.9 for 15 percent DCM-pH 7, and 12.4 for 100 percent DCM. These represent ranges of 93-107 for the former and 88-112 percent recovery for the latter case.

The ANOVA on the composite PCB-1260 results yielded only one significant term, the solvent-pH interaction term. The smallest deviations were for 15 percent DCM in hexane at neutral to basic conditions, which were not significantly different from the 100 percent DCM results at acidic to neutral conditions. With appropriate adjustment of the pH, either solvent system could be used, but the 15 percent DCM in hexane at pH 7-10 is the recommended choice. The rationale is that this system has been frequently used for extraction of these compounds in previous studies and is more often favored for the other groups in this study. Thus, there is consistency obtained by electing this solvent system.

The average deviation for the recommended extraction conditions was 1.9 percent recovery, which indicates that under these conditions one could expect recoveries from 98 to 102 percent on average.

For PCB-1221 both pH and solvent system showed significant effects on the percentage recovery of the spike. With respect to solvents, better recovery was obtained using 100 percent DCM. At neutral conditions, the results were considerably better (10 percent recovery) than they were at either pH 2 or 10. The interaction term was not significant, and therefore the recommended conditions are 100 percent DCM-pH 7.

The average recovery for any of the six peaks used in the analyses varied from 100 percent by 5.6 percent, indicating that recoveries of 94 to 106 could be expected on a routine basis.

For PCB-1232, the results were quite variable and no immediate answer was obtainable. By relaxing the required significance level to 10 percent, the pH-solvent interaction term does show significance and this result is more realistic, given the data as they exist. The best combinations were with 15 percent DCM at neutral to basic conditions or with 100 percent DCM at acidic to neutral conditions. This is a similar result to that obtained for PCB-1260, and the 15 percent DCM in hexane-basic conditions are selected for the same reason.

The deviations from 100 percent recovery average 8.8 percent, and the tendency is for these to fall below 100 percent. On average, then, a recovery level for this PCB of approximately 92 percent can be expected when the extraction is performed under these conditions.

The PCB-1242 results yielded no significant differences for any of the factors in the ANOVA. For this compound, either solvent can be used to obtain equivalent results and no pH adjustment is warranted. The deviation from 100 percent averaged 3.5 percent, and no strong preference was noted for the recoveries to be above or below 100 percent. The recoveries for each of the studied peaks, then, can be expected to be between 96 and 104 percent on the average.

Similarly, PCB-1248 showed no significant effects of pH or solvent, and no interactive effect between them. As such, either solvent system may be effectively used and no adjustment of the pH of the water is necessary. There was a slight preference for this PCB for the use of 15 percent DCM in hexane, but not of large enough magnitude to be able to show statistical significance.

The average deviation was 6.1 percent recovery, and the tendency was for the values to be lower than 100 percent. As a result, the expected recovery of this compound is approximately 94 percent, based on the seven peaks studied in this investigation.

Summary--

The overall best set of conditions for extraction of the pesticides and PCB's studied was to extract with 15 percent DCM in hexane at a basic pH. The recommended set of conditions for the single-compound pesticides, multi-compound pesticides and PCB's are shown in Table A12. From this, it can be observed that 100 percent DCM was the preferred solvent only for PCB-1221. Extraction at an acidic pH showed slight improvements for some of the single-compound pesticides and for toxaphene, but not enough to warrant a recommendation for acidic conditions. Neutral to basic conditions were favored for the single-compound pesticides and for four of the PCB's, and marked difference was observed with two of the single-compound pesticides. The mean recoveries which were obtained under these conditions are shown in Table A13 for all single-compound pesticides.

For the multi-compound pesticides and PCB's, the average deviation from 100 percent for the selected conditions is shown in Table A 14. These in several cases represent a plus or minus figure, where there were recoveries in excess of 100 percent obtained. This was the case for chlordane, toxaphene, and PCB's 1254, 1260, and 1242.

Preservation Study

Procedures and Results--

The preservation study was conducted to determine the effects of a seven-day storage period, at various conditions, on the recovery of the 25 substances of interest from dosed water samples. Each sample consisted of one quart of water dosed with one of the eleven groups, as in the extraction study. Two replicates for each of twelve conditions of pH, temperature and residual chlorine were prepared for each group as shown in the following model:

TABLE 5. EXPERIMENTAL MODEL FOR PRESERVATION STUDY

	4°C		*24°C*	
	0 ppm Cl	2 ppm Cl	0 ppm Cl	2 ppm Cl
pH2	2	2	2	2
pH7	2	2	2	2
pH10	2	2	2	2

The 2 ppm residual chlorine level was obtained, where required, by adding 160 microliters of Mallinckrodt sodium hypochlorite analytical reagent (5 percent minimum available Cl). This quantity of hypochlorite solution was determined by preparing a standard chlorine solution which was diluted to five different concentrations that were used to prepare an absorbance versus concentration curve[3]. Storage containers were one quart, flint glass, round, narrow-mouth bottles closed with aluminum foil-lined caps. Aluminum foil-lined caps were chosen instead of Teflon-lined caps in view of the experience of Bellar and Lichtenberg[4]. A sample was prepared by filling the bottle about two-thirds full with pH-adjusted water, adding 160 microliters of sodium hypochlorite (when required), swirling vigorously, adding the 100 microliters of dosing solution into the vortex, and adding the remainder of the pH-adjusted water while that already in the bottle was still swirling. The bottle was capped immediately and stored in a closed cardboard box at 4°C or 24°C. Care was taken not to slosh the bottle contents onto the aluminum lining of the cap after closure.

After storage, samples were extracted and analyzed as in the extraction study. The extracting solvent used was 15 percent DCM in hexane and extraction was performed at the storage pH, despite the indicated preference for 100 percent DCM and a particular pH in a few instances in the extraction study. These preferences were not considered to be of such magnitude as to override other factors, such as time, convenience, possibility of additional contamination being introduced, etc., which would be involved in meeting these preferences.

Data obtained in the preservation study are given in Tables A15 through A25. The data presented are uncorrected for values obtained for blanks at each of the 12 test conditions because of the variation in these values. However, major effects of storage may be noted when the results of the preservation study are compared with the extraction study results or when comparisons are made of the results at the various conditions in the preservation study (intratable comparisons). Some of these major effects are as follows:

α-BHC and δ-BHC	Large losses of these pesticides occurred at two or more sets of conditions.
Heptachlor epoxide	The amount of heptachlor epoxide indicated after storage increased by a factor of 1.5 to 2, or more, over the starting amount in a number of instances.
γ-BHC, heptachlor, aldrin, and endrin	Concentrations of these pesticides underwent drastic changes at certain conditions.
Endosulfan I, II	These disappeared at all conditions at pH 10.
Endosulfan sulfate	Only an estimated 5 to 10 percent of the starting amount of this compound remained after storage at all conditions.
Endrin aldehyde	This compound disappeared in the presence of chlorine at pH 10.

Chlordane	Large depletions of peak 2 occurred at some of the test conditions.
Toxaphene	Substantial depletions and increases affecting peaks 1 through 5 occurred at some of the test conditions. General shape of the chromatogram of this substance changed.
PCB-1221	At pH 7, 24°C, in the absence of chlorine, peaks 1, 2, and 4 were depleted 70 percent or more.
PCB-1232	At pH 7, 24°C, and in the absence of chlorine, peaks 1 and 2 were depleted 75 percent or more.

Figures A35 through A40 are chromatograms which show some effects noted immediately above. Chromatograms of standards of all of the groups are shown in Appendix A.

It was also noted that the presence of chlorine during storage often resulted in some broadening of the "solvent" peak during gas chromatographic analysis. Perhaps the worst case of broadening experienced is shown in Figure A41, where the broadening obscures the first peak being followed in the course of analysis.

The results of the preservation study were submitted to statistical analysis to determine the optimum storage conditions for each group and to estimate the recovery that could be expected for each compound under study. An ANOVA was used according to the model:

$$Y_{ijkl} = \mu + \rho_i + \tau_j + \gamma_k + \rho\tau_{ij} + \rho\gamma_{ik} + \tau\gamma_{jk} + \rho\tau\gamma_{ijk} + \epsilon_{ijkl}$$

where Y_{ijkl} = observed recovery for pH i, temperature j, chlorine level k, and repetition l
μ = overall mean
ϱ_i = effect of pH i, i = 1,2,3
τ_j = effect of temperature j, j = 1,2
γ_k = effect of chlorine level k, k = 1,2
$\varrho\tau_{ij}$, $\varrho\gamma_{ik}$, $\tau\gamma_{jk}$ = two factor interactions
$\varrho\tau\gamma_{ijk}$ = three factor interaction, and
ε_{ijkl} = random error associated with Y_{ijkl}

All terms (pH, temperature, chlorine) are considered fixed effects, and all F-ratios calculated against the error mean square. Each compound is investigated separately and the results synthesized to obtain a recommended set of conditions for storage.

As in the extraction study, the interaction factors are to be interpreted as an instance where the recovery obtained was better (worse) than could be expected by observation of the average results for pH, temperature, and/or chlorine taken individually. The conclusion is that the means of the combinations should be used to provide the estimates of percent recovery, and no physical interpretation is implied. The precision of the analyses allowed, in some cases, relatively small differences to be termed significant, while there may be no practical value to the difference between average recoveries in these cases.

The synthesis of the results was carried out in the following manner. For each single compound pesticide and for each numbered peak in remaining materials a separate ANOVA was conducted and the optimum conditions were determined. From these, candidate storage conditions were obtained and evaluated against each other. The mean recoveries were broken out for each set of conditions and the selection was then based on which of the candidates provided the best recoveries for all components taken as a group.

Single-Compound Pesticides--There were marked effects on certain of the single-compound pesticides upon preservation. In Table A26 and A27 are the ANOVA summaries for these pesticides, with the compounds split according to the analytical groupings used for chromatography. Endosulfan sulfate was not detected in the preserved samples, and recoveries as high as 269 percent were obtained for heptachlor epoxide in the same water samples. The high recoveries for heptachlor epoxide indicate that an interference occurred, perhaps from the degradation products of the endosulfan sulfate, and these data were not deemed suitable for data analysis. Although some of the recoveries were very near 100 percent, it is impossible to determine whether this represented complete recovery of the compound itself or partial recovery of a combination of desired compound and interfering substance.

In the second group, endosulfan I and II were lost completely on storage at pH 10, and endrin aldehyde was lost in the presence of residual chlorine, and the data analysis was restricted to pH's 2 and 7. In nine of the 14 compounds evaluated, significantly better recoveries were obtained when the samples were stored at 4°C. The pH-temperature interaction was significant in several cases, and indicated that pH 2-4°C and pH 7-4°C were the most favored conditions for storage. By inspection of the mean recoveries, the best set of conditions appears to be pH 7-4°C for the single-compound pesticides. These conditions result in recoveries for α-BHC and aldrin of 60 and 65 percent, respectively, but are equivalent to or better than the pH 2-4°C results. Some improvement could be obtained if only one of these was suspected to be in the water, where an alternative set of conditions could be used.

The only marked effect of residual chlorine is in the aldrin data, where the recovery was only 15-20 percent. There were some differences noted for other compounds, but only when the samples were stored at 24°C, which is not recommended. The conclusion is that aldrin is the only one of the single-compound pesticides studied for which storage would not be allowable in the presence of residual chlorine under the conditions recommended here.

Chlordane--For chlordane, the results of the ANOVA's on the six numbered peaks measured are contained in Table A28. The storage temperature was a significant term on the first three peaks, and the pH-temperature interaction was significant on peak 3.

In all three instances where temperature was significant, storage at 4°C was indicated to give better results. The pH-temperature interaction means were separated using Fisher's LSD test, and the results were equivalent for all combinations except pH 7-24°C and pH 10-24°C, which were somewhat lower. As a result, the recommended storage conditions for chlordane are to store at 4°C, with no requirement for adjustment of the pH of the water.

The average recoveries for the numbered peaks in chlordane are shown at the bottom of Table A28. Peaks 4 through 6 gave recoveries in excess of 100 percent, but all conditions were equivalent. In none of the studied peaks did the presence of residual chlorine interfere with the results.

Toxaphene--The results of the ANOVA's on the six numbered peaks are shown in Table A29. The pH of the water was a significant term for five of the six peaks. On four of these, the optimum conditions were at pH's of 2 and 7, indicating a preference for nonbasic conditions. Two of the peaks, 1 and 5, varied according to the storage temperature, both indicating better preservation at 4°C. The pH-temperature interaction was the only interaction to indicate significance for toxaphene, where for peak 1 the best combinations were pH 2-4°C and pH 7-4°C.

The results for peak 2 were not readily interpretable. The pH factor gave a mean recovery of 100.3 for pH 10, with recoveries of 120.5 and 118.8 for pH's 2 and 7, respectively. Upon examination of the pH-temperature interaction, however, the recoveries ranged from 114 percent at 4°C to 86 percent at 24°C, and clearly indicate a problem was existent. The high values seem to indicate that peak 2 is not a reliable indicator of the concentration of this compound, and consideration must be given to deleting this peak from the compound identification or quantification set.

The recommended storage conditions for this compound, then, are pH 2 to 7 (nonbasic) and 4°C. Recommended peaks for use in quantifying are 1 and 3-6. Mean percent recoveries for this set of conditions are shown in Table A29. No effect of residual chlorine was noted in any of the studied peaks.

PCB-1254--The summary of the ANOVA's is shown in Table A30 for the substance under study. Principal factors of significance were the storage temperatures for four of the seven peaks studied. In each of these cases, the preferred temperature was 4°C. In one case, the pH was determined to be significant for peak 5. The highest recovery was 102.3 for pH 7, which was statistically indistinguishable (using an LSD statistic) from the result for pH 2. However, the mean recovery for pH 10 was 95.1, which is obviously an acceptable result.

The pH-temperature interaction was significant for the ANOVA on peak 7 alone. The separation of these means was incomplete, however, and the results did not provide a meaningful answer. On the basis of the previous peaks, the indication was that storage at 4°C would be recommended. The mean recoveries for the three pH's at 4°C were indistinguishable from each other, indicating that no consideration of pH is necessary for this peak.

The recommendation for this PCB is that samples be maintained at 4°C with no necessity for adjustment of the existing pH. The mean recoveries for the numbered peaks are shown in the table, and all can be considered good indicators of the quantity of the substance present. There was no influence on the results from the presence of residual chlorine.

PCB-1016--The summary of the ANOVA's on the preservation study results is shown in Table A31 for PCB-1016. The principal results obtained from this summary were that cold storage was preferred for three of the eight numbered peaks. Only one peak showed a better result dependent upon the pH of the sample, where neutral conditions were favored. The interaction terms provided little additional information. The combination of pH 7-4°C was equivalent to the best conditions for all cases, and the pH-chlorine term was not important to these results. The recommended conditions, then, are adjustment to pH 7 and storage at 4°C. The mean recoveries for each of the peaks are shown at the bottom of the table.

As can be seen, the recovery for peak 5 was 111 percent on average, with no significant differences due to conditions. Some consideration must be given to deleting this peak as an identifier for this group. The remaining seven numbered peaks provide realistic, and self-consistent recoveries, and should be adequate for identification.

The effect of chlorine on these samples was minimal. For peaks 2 and 4, the temperature-chlorine interaction indicates that less than optimum results were obtained at 4°C in the absence of chlorine. However, for peak 2 the recovery was 93.5 percent under these conditions and for peak 4 an average of 90.3 percent was observed, both quite acceptable results. For this group, then, no substantial effect from residual chlorine in the sample can be expected when the storage conditions above are employed.

PCB-1260--The summary of the ANOVA's on the PCB-1260 results is shown in Table A32. For these data, the primary factor for consideration was the pH of the water, giving significant differences on six of the eleven numbered peaks in the chromatogram. Temperature was a significant factor for four of the peaks. The pH indicated was either pH 2 or equivalent between pH's 2 and 7 for all instances where a difference occurred. Similarly, the temperature differences all favored 4°C. The interaction terms did not indicate any discrepancy from these results, indicating that the pH 2-4°C combination was equivalent to the best recovery for both cases where pH-temperature factors were significant. The conclusion is that water samples for this PCB should be maintained cold after adjustment to pH 2.

No effect of residual chlorine in the water was noted for these results. Neither the chlorine nor the pH-chlorine interaction resulted in any significant factors. The temperature-chlorine interaction was significant only on peak 9, and for these data, the optimum conditions were 4°C-0 ppm chlorine and 4°C-2 ppm chlorine, meaning that no difference was observed at the selected temperature for storage.

The mean recoveries for the storage conditions are shown in Table A32 for each of the eleven numbered peaks in the chromatogram. All represent good recovery and the values are reasonably self-consistent.

PCB-1221--The results of the ANOVA's on PCB-1221 are summarized in Table A33. As can be seen, these varied considerably with the storage conditions. The favored pH's were 2 and 10 for three peaks, 7 and 10 for one, and indeterminate for two others. The indeterminates were cases where the average result contained gross discrepancies based upon the combinations of pH and temperature which made a simple comparison infeasible. Storage temperature at 4°C was significantly better for five of the seven numbered peaks.

The interaction terms provide the information which dictates the choice of storage conditions. The combination of pH 2-4°C was determined to be among the lowest average recoveries for three of the peaks, and to provide lower results in the three-factor interaction twice, once in the presence of chlorine and once in the absence. The pH 7-4°C combination was disfavored once, especially in the presence of chlorine, and also does not appear to be a viable candidate. The choice, then, is pH 10-4°C, which shows to be an acceptable result for all peaks under all conditions. The mean recoveries for this combination are shown at the bottom of Table 33.

Chlorine in the water did affect the results for this PCB. However, there was no difference detected between the results at pH 10-4°C dependent upon the residual chlorine, and if these storage conditions are used residual chlorine should represent no problem in the preservation for this substance.

PCB-1232--The results for PCB-1232 are summarized in Table A34. Similar to Group VIII, there were large differences which resulted from the variation in the storage conditions. Where pH differences existed, the tendency was for 7 and 10 to be favored, and 4°C was the favored temperature on the six peaks where a difference was noted. The interaction between pH and temperature gave consistent results, with pH 7-4°C and pH 10-4°C being the most frequently preferred conditions. The means for both of these combinations are shown at the bottom of Table A34.

The better choice between the two is the pH 10-4°C combination, based on the results from peak 1 primarily. The mean recoveries are fairly self-consistent, and only the peak 7 results give cause for concern. These values were above 100 percent in many instances and may not truly represent the concentration of the PCB of interest.

The chlorine in the samples does not provide an interference with the conditions selected for storage, primarily resulting in problems at pH 7 and at 24°C. For the three peaks for which the three-factor interaction was significant, the average recoveries were equivalent at pH 10-4°C.

PCB-1242--The ANOVA's on this PCB are summarized in Table A35 for the nine numbered peaks used for identification. The pH of the water was the most important factor, giving significant differences on eight of the nine analyses. There was no consistency, however, with all three pH's used being best for certain of the peaks. The storage temperature was significant for two of the peaks, with 4°C providing the better results in both cases. From the pH-temperature interaction terms, the candidates for storage are unable to be further reduced, with pH's 2, 7 and 10 at 4°C all appearing as superior combinations. The means for all nine peaks under the three sets of conditions are shown at the bottom of Table A35.

The most consistent results are those for pH 2-4°C, and this is the recommended set of conditions. However, storage at 4°C with no adjustment of the pH would provide acceptable data.

There is some chlorine effect in these data, with the first three peaks having better results when there was residual chlorine present. However, the mean recoveries for these three peaks showed only 2-3 percent recovery differences, which has little practical significance. For the remaining peaks, no difficulty is indicated for the recovery of this PCB in the presence or absence of residual chlorine in the sample.

PCB-1248--The summary of the ANOVA's on the seven numbered peaks used for identification of PCB-1248 is contained in Table A36. For this group, few differences were noted in the data resulting from the storage conditions, with the majority of the significant differences occurring in the interaction terms. From peak 1, candidates of pH 2-4°C and pH 10-4°C occur, with pH 7-24°C being eliminated on the basis of the interference noted for pH 7 in the presence of residual chlorine. For peak 2, both of these are equivalent, but pH 2 in the absence of chlorine resulted in less acceptable results. As a result, pH 10-4°C is the recommended set of conditions for storage. The mean percent recovery observed for each numbered peak is given at the bottom of Table A36 for these conditions.

The presence of residual chlorine presented no problems in the storage for this particular PCB under the recommended conditions. For the two peaks (4 and 5) where the temperature-chlorine interaction was significant, the results were equivalent at pH 10.

Summary--

The optimal conditions determined for the pesticides and PCB's studied are shown in Table A37. From these a recommended set of conditions for the category as a whole can be made. The necessity for cold storage is obvious, but the question of an adjustment of pH is open since there were inconsistent recommendations for the various materials. The overall recommendation is that the water be adjusted to pH 2, based primarily on the single-compound pesticide results where two of the pesticides were not detectable after seven days storage at pH 10. The mean recoveries which can be expected upon analysis after storage under these conditions are given in Table 6 for the substances under study.

Where a particular compound is of interest, the analyst may be able to increase the recovery by selecting a set of conditions specifically for that compound or material.

TABLE 6. MEAN PERSERVATION RECOVERIES
(pH 2-4°C)

Material	Recovery
α-BHC	64
β-BHC	86
δ-BHC	80
Heptachlor epoxide	*
DDE	94
DDD	98
DDT	94
Endosulfan sulfate	+
γ-BHC	100
Heptachlor	82
Aldrin	89 (15)#
Endosulfan I	96
Dieldrin	105
Endrin	92
Endosulfan II	102
Endrin aldehyde	88
Chlordane	80-110**
Toxaphene	93-113
PCB-1254	95-102
PCB-1016	91-111
PCB-1260	90-101
PCB-1221	85-115
PCB-1232	82-105
PCB-1242	90-101
PCB-1248	92-93

* —Interferences prevent analysis
+ —Disappeared under all conditions
# —If residual chlorine present
** —Range of recovery for various peaks studied

SUMMARY OF PHASE I

With the exception of endrin, all substances of interest in this program can be chromatographed on the two columns selected for use in this work. Column 1 (1.8m × 6.35mm glass tubing packed with 1.5 percent SP-2250 plus 1.95 percent SP-2401 on 100/120 mesh Supelcoport) is preferred over Column 2 (same as Column 1 except the liquid phase is 3 percent OV-1) because 16 pesticides can be chromatographed in two groups on it, a feat that cannot be done with Column 2. Also, Column 2 is unsatisfactory for endrin.

Two detectors were selected for evaluation in the program: (1) a Hewlett-Packard linear Ni^{63} electron capture detector and (2) a Tracor 310 Hall electrolytic conductivity detector. The former is sensitive to picogram quantities, whereas the latter is sensitive to nanogram quantities. The Hall detector is more selective for organochlorine compounds, a feature which is potentially valuable in the presence of interferences such as phthalates and polynuclear aromatic hydrocarbons.

Clean well water was dosed with a quantity of substance inversely related to the detector response for the substance. Thus, α-BHC was spiked at the 20 parts-per-trillion level and toxaphene at the 4 parts-per-billion level. Other substances were dosed at levels in between these extremes. Extraction was performed with 15 percent dichloromethane (DCM) in hexane and with 100 percent DCM at pH's of 2, 7 and 10. The most serious problem encountered was the presence of interfering peaks in chromatograms of blanks. The magnitude of these peaks varied so subtraction of a constant blank was usually not possible. Occasionally, work had to be repeated. Recoveries were usually high except for heptachlor and aldrin. A statistical analysis of the data is presented. Although minor differences were noted in extraction efficiencies, the differences are not great enough to give substantial reasons for choosing either solvent in preference to the other or a particular pH at which to do the extraction.

A preservation study was conducted to determine the effects of a seven-day storage period at twelve conditions of pH, temperature, and residual chlorine level. A statistical analysis of the data is given. A number of deleterious effects were observed for about half of the 25 substances. However, 23 of the substances can be stored for one week at 4°C, under neutral or acidic conditions, and in the presence or absence of two parts per million chlorine with the only losses suffered being of a tolerable magnitude. Other conditions offer minor improvements in the losses incurred, as pointed out in the statistical analysis. Aldrin, at the study level, cannot be stored in the presence of chlorine at acid or neutral pH's without a 75-90 percent loss. Endosulfan sulfate, at the study level, was lost to the extent of 90 percent or more at all conditions.

SECTION 5

PHASE II

OBJECTIVES

The objectives of Phase II were as follows:

1. Investigation of two liquid-solid chromatographic cleanup procedures, including the determination of the separation patterns and recoveries for all of the substances of interest.
2. Application of the method to five wastewater samples from industrial discharges. This aspect of the work included modifications of the method as required to analyze the wastewaters. These modifications would be based on experience gained earlier in the program and on new developments reported in scientific literature.
3. Generation of accuracy and precision data for all substances of interest by dosing them into the five wastewaters. Determinations would be made immediately after dosing and after one week of storage at the best conditions observed in Phase I. It was hoped that these dosing and recovery experiments would permit the assignment of detection limits for each substance in the wastewaters.

PROGRAM DIRECTION

Representatives of SwRI met with the project officer in Cincinnati before Phase II work commenced. Discussion with respect to Phase II was primarily on the cleanup columns and procedures to be used. Florisil and alumina were selected as the adsorptive solids. Fractionation of compounds was limited to that which can be obtained on the above-named solids. Fractionation on secondary columns such as silica gel or charcoal was considered to be beyond the scope of this program.

EXPERIMENTAL

During Phase I work it was noticed that for endosulfan sulfate a lower than expected value for the amount which should have been present was occasionally observed. At first this was attributed to a general instability of the compound. However, a closer look into this problem during Phase II revealed that endosulfan sulfate instability was related to acetone solutions. In hexane, no loss was measured in a two-months period at 4° or 24°C. In methyl alcohol, the stability was comparable to that in hexane. In acetone at 4°C, the concentration was only 33 percent of the original after two months. In acetone at 24°C, the concentration was only three percent of the original after three weeks.

In Phase II work, endosulfan sulfate was always prepared and dosed in a hydrocarbon solvent. Since dosing with a hydrophilic solvent had been considered preferable at the beginning of Phase I, this compound could have been dosed in methyl alcohol during Phase II except for two reasons: (1) the information that it was stable in methyl alcohol was not available until Phase II was several months underway, and (2) a source of the pure solid material was unknown until after Phase II began. The only source of supply up that point was as a hydrocarbon solution. All other Group I components were dosed in acetone, as was done in Phase I.

Liquid-Solid Column Chromatography

Florisil--

The Florisil cleanup column procedure incorporates features of methods published earlier[5,6,7]. The column was prepared by gently packing Florisil PR, which was taken directly from storage at 130°C for a minimum of 24 hours, to a height of 10cm (about 21 grams) in a 400mm × 25mm (OD) glass column containing a coarse fritted disc and fitted with a Teflon stopcock. A layer of sodium sulfate 1-2cm in height was added. The column was prewetted with 60mL of hexane, drained to the top of the sodium sulfate, then a 5mL volume of hexane containing a dose of one of the groups was put on the column which was drained again to the top of the sodium sulfate. Elution was then performed using 200mL volumes of 6, 15 and 50 percent ethyl ether in hexane, receiving each 200mL elution fraction in a Kuderna-Danish flask. Each fraction was concentrated to a 10mL final volume and an appropriate volume was injected into the gas chromatograph.

The evaluation of this procedure consisted of preparing for each substance 5 columns that were identically dosed and eluted, followed by analysis of the recovered fractions.

The quantity of substance placed on the column in hexane was keyed to the specific response of the substance to electron capture detection, starting with 1μg (equivalent to one part per billion in 1L of water) for the substances giving highest response and ranging upward to 200μg for toxaphene. The quantities added for the 25 substances are shown in Table 7.

TABLE 7. QUANTITIES ADDED TO COLUMNS IN EVALUATION STUDIES

Compound	μg
Aldrin	2
α-BHC	1
β-BHC	2
γ-BHC	1
δ-BHC	2
Chlordane	20
p,p' DDD	6
p,p' DDE	3
p,p' DDI	8
Dieldrin	3
Endosulfan I	3
Endosulfan II	5
Endosulfan sulfate	15
Endrin	5
Endrin Aldehyde	12
Heptachlor	1
Heptachlorepoxide	2
Toxaphene	200
PCB-1016	25
PCB-1221	55

(continued)

TABLE 7. (cont'd)

Compound	μg
PCB-1232	55
PCB-1242	28
PCB-1248	40
PCB-1254	40
PCB-1260	80

The distribution and recovery of the substances in the three fractions of eluate are shown in Table 8. A statistical analysis of the recovery data for each substance is given in Tables A38 through A48. These data show that recovery from a Florisil cleanup column is good. The lowest recovery was recorded for PCB-1254 at about 90 percent. Recovery percentages for the other substances were closer to 100 percent.

TABLE 8. DISTRIBUTION AND RECOVERY OF CHLORINATED PESTICIDES AND PCBs USING FLORISIL COLUMN CHROMATOGRAPHY

	Recovery (%) by fraction		
	1(6%)*	2(15%)	3(50%)*
Aldrin	100		
α-BHC	100		
β-BHC	97		
γ-BHC	100		
δ-BHC	98		
Chlordane	100		
(p,p') DDD	99		
(p,p') DDE	98		
(p,p') DDT	100		
Dieldrin	0	100	
Endosulfan I	37	64	
Endosulfan II	0	7	91
Endosulfan sulfate	0	0	106
Endrin	4	96	
Endrin aldehyde	0	68	26
Heptachlor	100		
Heptachlor epoxide	100		
Toxaphene	96		
PCB-1016	97		
PCB-1221	97		
PCB-1232	95	4	
PCB-1242	97		
PCB-1248	103		
PCB-1254	90		
PCB-1260	95		

* % Ethyl ether in eluant

During the early work, it was assumed that the Florisil activity would have to be checked periodically. This was done by applying a mixture of heptachlor epoxide, dieldrin, and endosulfan II to the column and eluting as described. All of the heptachlor epoxide should be in the first fraction, all of the dieldrin in the second, and 90 percent or more of the endosulfan II in the third fraction. However, this procedure proved to be unnecessary so long as the Florisil had been stored at 130°C for 24 hours or longer. The activity remained constant, even when going from one lot or batch to another. Not having to continually check the activity of the Florisil is one of the advantages accruing to the use of the fully activated material as versus the use of the partially deactivated material.

Alumina--

The alumina column cleanup procedure which follows is based on the method of Telling, Sissons, and Brinkman[8].

Add 10mL of water to 90 grams of alumina (Woelm N-Super I), as purchased, in a jar, cap the jar, shake until smooth flowing, then let stand 24 hours before using. Slurry 22 grams of the prepared alumina in enough hexane so it can be poured into a glass column (400mm × 25mm OD coarse fritted disc, Teflon stopcock). Settle the alumina with gentle tapping on the glass column and add a layer of sodium sulfate 1-2cm in height. Wash the column with 20mL of hexane and add the dosed hexane which should be about 5mL in volume. Elute, successively, with 40mL hexane, 110mL hexane, 100mL 50 percent ethyl ether (containing 2 percent ethyl alcohol) in hexane, receiving each fraction in a Kuderna-Danish flask. Concentrate the fractions to final volumes of 10mL and inject appropriate quantities into the gas chromatograph.

The evaluation of this procedure consisted of preparing for each substance five columns that were identically dosed and eluted, followed by analysis of the recovered fractions.

The quantities of substances applied to the column were the same as those applied to the Florisil column (Table 7). The distribution of the groups of substances of interest in the three fractions is shown in Table 9. A statistical analysis of the recovery data for each substance is given in Tables A49 through A59. The lowest recoveries were experienced with DDT at about 93 percent and endosulfan II at about 86 percent.

Evaluation of Results--

Recoveries from both columns are excellent. Elution times with the alumina column are shorter than those with the Florisil column. However, the single compound pesticides, chlordane, and toxaphene are more frequently found in two fractions when using the alumina column than with the Florisil column. Thus, processing and analyzing additional fractions, as would be the case with alumina, would likely be more time consuming than the longer elution times with Florisil. Since more components are recovered near the 100 percent level in the first fraction (Florisil), this advantage appears to more than offset the less significant advantages of the alumina. Also, Florisil may be stored, as received, in a 130°C oven for indefinite periods of time and still be usable. The alumina, as received, must be deactivated to Activity IV (10 percent water, by weight) in a careful manner, then protected with care until used. How long Activity IV alumina may be held before use is not known. However, it appears to be a reasonably long period, such as two weeks or longer.

Wastewater Application

With the assistance and approval of the project officer, five wastewaters were procured and analyzed. The sources of these wastewaters and the results of the analyses are given below. All samples were put into 1-gallon glass bottles which had previously contained Burdick and Jackson "distilled in glass" solvents, suitable for pesticide residue analysis. Sample sizes

TABLE 9. DISTRIBUTION AND RECOVERY OF CHLORINATED PESTICIDES AND PCB's USING ALUMINA COLUMN CHROMATOGRAPHY

	Recovery (%) by Fraction		
	1 (40mL Hexane)	2 (110mL Hexane)	3 (100mL 50% Ethyl ether in Hexane)
Aldrin	97	2	
α-BHC	27	69	
β-BHC		15	87
γ-BHC			98
δ-BHC		103	
Chlordane	Spread through first two fractions mostly; traces in third fraction; average total recovery—98%		
DDD (p,p')	16	83	
DDE (p,p')	91	6	
DDT (p,p')	82	11	
Dieldrin		104	3
Endosulfan I		91	3
Endosulfan II			86
Endosulfan sulfate			102
Endrin		105	
Endrin aldehyde			105
Heptachlor	93	6	
Heptachlor epoxide		99	
Toxaphene	Spread through first two fractions; average total recovery - 97%		
PCB-1016	99		
PCB-1221	99		
PCB-1232	98		
PCB-1242	98		
PCB-1248	98		
PCB-1254	100		
PCB-1260	100		

ranged from 20 to 24 gallons. All samples were shipped by air, unrefrigerated. Time in shipment was five days for Wastewater 1 and about two days for the others. Upon receipt at SwRI, the samples were pooled in a 25-gallon stainless steel vessel, the pH adjusted to 3-4 (if required) with concentrated sulfuric acid, returned to the bottles in which they were shipped, and stored at 4°C until used.

Analyses of Wastewaters--

In order to develop method improvements and to provide base data for the dosing and recovery experiments and the accuracy and precision evaluations to follow, each wastewater was analyzed in triplicate for each substance of interest in this program. Hence, tables of data are presented as indicated below for each wastewater. The values given in the tables are from the computer printout sheets and result from integration of the areas of peaks appearing in the retention windows. The fact that the peaks are in the retention windows does not mean that they coincide precisely with the peaks from standards. As an arbitrary working device and to allow for small errors attendant to injection, flow control, temperature control, etc., coincidence of retention times was considered for identification purposes to be anything within 0.03 minutes.

Wastewater 1--This wastewater was sampled by personnel of the Surveillance and Analysis Division, USEPA, Athens, Georgia and was identified as HI-003 by the Athens office. The pH as received was between 7.5 and 8. The wastewater was light yellow to light orange in color, free from solids and had a mild odor reminiscent of pine oil plus a sweet overtone. The plant being sampled produces toxaphene and other organic materials. Our understanding is that this effluent was from all of the plant operations other than the toxaphene unit. The toxaphene plant effluent treating unit was "down" on the day of sampling, and therefore no effluent was being discharged from it at that time.

As a preliminary step to the analysis of Wastewater 1, extracts obtained with 15 percent DCM in hexane (no emulsions) were cleaned up with Florisil and alumina. Of the three fractions taken from each column, alumina produced the cleaner first two fractions but a much dirtier third fraction. Since six single compound pesticides are determined in the third fraction from the alumina but only two in the third fraction from the Florisil, Florisil was the better overall choice and it was used in all of the work with Wastewater 1. The extracting solvent was 15 percent DCM in hexane. Three replicates of one liter each were extracted, cleaned and fractionated as in the liquid-solid column chromatography study. These fractions were chromatographed and analyzed against the appropriate standards for the fractions. Typical chromatograms of the three fractions are shown as Figures A42, A43 and A44. Tables A60 through A70 give the analytical results. Comparison with standards rules out all of the pollutants of interest in this program except heptachlor and endosulfan II and PCB-1242. The presence of PCB-1242 is more questionable than the other two substances, since only six of the nine peaks being followed in the analysis of this substance are present, and the amount present, as indicated by each peak, is not a constant value. This is strong but not conclusive evidence that PCB-1242 is not present. However, if the substance present is PCB-1242, the amount indicated is less than 2 parts per billion, and the other two substances are at 20 and 40 parts per trillion, respectively.

Wastewater 2--Wastewater 2 was sampled by personnel of the Eastern District Office, USEPA, Westlake, Ohio. No information was supplied as to the source or character of this sample. The sample as received was amber in color, free from solids, had a urine-like odor, and was at pH 4.

Extractions were performed with 15 percent DCM in hexane, and no emulsion problems occurred. Florisil was the cleanup adsorbent. Alumina ws tried but was not advantageous.

Chromatograms of the three fractions from a one-liter sample of this wastewater are shown in Figures A45, A46 and A47. Analytical data, taken from computer printout sheets, for three replicates of one liter each are given in Tables A71 through A81. Comparison of retention times with standards rules out the presence of any of the pollutants of interest in this program.

Despite the absence of substances of interest, the substances which were present provided many interferences in the spiking and recovery experiments (see subsequent section, accuracy and precision). Due to the interference problems, especially from the substances eluting from the GC column during the first four minutes, a considerable effort was made to identify the interferences, with the hope that identification would suggest approaches to their removal. The presence of elemental sulfur was strongly suspected. Treatment of first fractions of extracts with elemental mercury[9] was commenced, but the results were erratic. Careful drying of the extracts, addition of a mercuric salt, addition of acid-washed copper[10], and prolongation of the agitation period were without consistently beneficial effects.

During concentration of the extracts in the Kuderna-Danish apparatus, a deposit of yellowish needle crystals and a gummy residue were noted. Infrared spectroscopy examination suggested that pentachlorophenol might have been present.

GC/MS analysis of the concentrated extract indicated the presence of naphthalene, methyl naphthalene, two dichlorinated compounds, 2-methylthiobenzothiazole, phthalates, dioctyl adipate, anthracene, and two hydrocarbons. No pesticides were found.

Wastewater 3--Wastewater 3 was procured by SwRI-Houston personnel. It was the final chlorinated effluent from the Northside Treatment Plant in Houston. This plant treats both residential sewage and industrial plant effluents. This wastewater as received had a mildly bad odor, a cloudy appearance, and a pH near 7.

Preliminary extractions were performed with 15 percent DCM in hexane and 100 percent DCM. Bad emulsions were encountered with both solvents, but the ones with 15 percent DCM in hexane were more manageable and hence this solvent was used, along with a Florisil column cleanup.

The emulsions were broken to a large extent by patiently waiting, adding additional solvent, "whipping" the emulsion layer with a Nichrome wire, and adding sodium sulfate when the amount of unbroken emulsion was small in volume.

Reduction of sulfur interference in first fractions was desirable with this wastewater and agitation with mercury was generally used. The results were erratic but less so than with Wastewater 2.

Figures A48, A49 and A50 are chromatograms of the fractions from a one-liter sample of Wastewater 3. The analyses of three one-liter replicates yielded the data in Tables A82 through A92. Comparison of retention times with standards suggests the presence of DDE at 22 parts per trillion, dieldrin at 38 parts per trillion and chlordane at about 600 parts per trillion. Although only two peaks are shown as matching with chlordane, they are the major peaks (4 and 5) for the substance. The lesser peaks might not be detectable in this matrix at this level. Five out of the ten peaks match the standard for PCB-1232 (Table A98) but the amount present, as indicated by each peak, is quite variable which is strong evidence against the presence of this substance. Therefore, PCB-1232 was not considered to be present.

Wastewater 4--Wastewater 4 was procured by SwRI-Houston personnel, also. It was from one of the influent streams to the Northside Treatment Plant in Houston, originating in a highly industrialized area of the city. As received, this sample had a rank sewage odor, some solid material, a cloudy appearance, and a pH near 7.

Extractions were made using 15 percent DCM in hexane. Florisil was used to clean up the extracts, followed by agitation of the first fractions with mercury. Emulsions formed as with Wastewater 3 and were treated as reported for that wastewater.

Treatment with mercury produced somewhat erratic reductions of interferences. Six replicates were treated. The last two were subjected to an increased amount of violent agitation with the vortex tube agitator as compared to the first four. This resulted in more uniform and lower analytical values as may be seen in Tables A93 through A103, in the data derived from first fractions. Figures A51 and A52 are first fraction chromatograms showing Replicate 1 and Replicate 5 (increased agitation with vortex tube agitator). Figures A53 and A54 are typical chromatograms of the second and third fractions of Wastewater 4. Comparison of retention times with standards indicates the presence of DDE at 31 parts per trillion, heptachlor at 119 parts per trillion, dieldrin at 32 parts per trillion, and chlordane at 1.4 parts per billion.

Wastewater 5--This sample was taken by Southern Research Institute personnel, through arrangements made by the EPA, from the final effluent stream from a pesticide manufacturing plant in Memphis, Tennesse. This effluent was clear, had a "chemical" odor, and had a pH around 3, as received.

Extractions were performed with 15 percent DCM in hexane. Emulsification was not a problem. Column cleanup was carried out with Florisil after a preliminary comparison with alumina showed no advantage resulting from the use of alumina.

Chromatograms of the three fractions from an extract of a one-liter sample are shown as Figures A55, A56 and A57. This wastewater had a very high background of substances which were electron capture sensitive compared to the other four wastewaters. However, most of the substances producing peaks are not substances of interest on this program. The analyses of three replicates produced the data shown in Tables A104 through A114. Comparison of retention times with standards indicates the presence of heptachlor at 6.6 parts per billion, heptachlor epoxide at 0.5 parts per billion, and endrin at 32 parts per billion. GC/MS examination of the first and second fractions of an extract indicated that nearly all of the substances are chlorinated but only the presence of heptachlor was confirmed.

Dosing of Wastewaters and Recovery Analyses--

In order to accumulate data for accuracy and precision evaluations, each group was put into six one-liter portions of each wastewater, one group at a time. Three of these dosed samples were analyzed immediately and three were stored for seven days in sealed, glass containers, in the dark, at 4°C. The dosed quantities were the same as those given previously in the liquid-solid column chromatography experiments, except where interfering background required that the quantity be increased. Such instances are noted on the data tables presented subsequently. Except where noted to the contrary, extraction, cleanup and GC analysis of the extracts were as given in earlier sections of this report.

As mentioned in the section on the analysis of the wastewaters, the data obtained resulted mostly from interferences, not chlorinated pesticides nor PCB's, falling within the retention window when computer analysis was employed. Restriction of this window to a practical minimum is helpful but not sufficient to prevent serious analytical problems. These interferences are so close to the retention times of substances of interest that the computer reads them as authentic substances of interest when the sample has not been dosed. However, when the sample has been dosed with authentic substances the computer may or may not be able to discriminate between the interferences and the authentic substances. Therefore, the peak integration data had to be constantly reviewed and evaluated as to whether the interfering background had or had not been combined with a particular peak. Maintaining total objectivity in these instances and attaining completely reliable readings were not always possible. It was not possible to obtain a rigidly reproducible set of background data which could always be subtracted from dosed sample analytical data. In many instances, data acquired through peak height measurements were more uniform and reasonable. Wherever possible, computer printout data were accepted as is. Where doubt existed as to the acceptability of the computer data, peak height measurements were made. If significant differences in the data were observed, the peak height data were always considered the more reliable and are presented in the subsequent tables.

The data presented in the tables referred to below were used to calculate a "Method Recovery", i.e., the percent recovered at zero time of what had been added and a "Preservation Recovery", i.e., the percent remaining after seven days of the total amount in the dosed wastewater at zero time. This was performed as follows:

let k = background (amount found in the undosed wastewater)
let y = quantity added to wastewater
let x_0 = quantity found in dosed wastewater at zero time for replicates 1, 2 or 3
let x_{0avg} = average quantity found in dosed wastewater at zero time
let x_7 = quantity found in dosed wastewater after 7 days for replicates 1, 2 or 3

Then

"Method Recovery" = $(x_0-k)100/y$ % (calculated for each replicate)

and

"Preservation Recovery" = $(x_7)(100)/x_{0avg}$ %(calculated for each replicate)

Toward the conclusion of work with Wastewater 3, a technical review meeting was held at Southwest Research Institute. The decision was made during this meeting to perform more experimentation on cleanup procedures, reducing the number of spiking and recovery experiments as required to accommodate this change in emphasis. The goal of this change was the development of alternative methods which would reduce interfering background caused by nonpriority pollutants. Such new methods would permit the analysis of certain priority pollutants, especially the early eluting ones, at a lower concentration than was initially possible in Wastewater 5, in particular.

As a result of this change in emphasis, only 8 single compound pesticides, chlordane, and PCB-1254 were dosed and recovered in work with Wastewater 4. This was done largely to reduce the overall workload of the program, in keeping with the decision mentioned above. This judgment was based on the fact that Wastewaters 3 and 4 were quite similar in character and problems. Since work with Wastewater 3 had been successful, there was no reason to suppose that work with Wastewater 4 could not be completed successfully also, but with minimal gain in new information. Hence, work was limited with this sample, as reported subsequently.

Work with Wastewater 5 was also limited so the experimentation reported below could be conducted.

Wastewater 1-The dosing and recovery experiments went smoothly with Wastewater 1. Tables A115 through A125 give the data obtained. It is estimated that the dosing quantities could have been reduced to 25 percent of the quantities used with analytical results remaining satisfactory in most instances. The greatest difficulties occurred with interferences for one or two early eluting peaks of PCB-1221 and PCB-1232. If these peaks had been eliminated from the analyses, adequate peaks would have remained for analytical purposes, and the ranges given for these substances would have been narrowed. Recovery ranges were as follows in Table 10.

TABLE 10. RANGES OF RECOVERIES, WASTEWATER 1

	Ranges of Recoveries, %, avg	
	Zero Time	**7-Days' Storage**
Single compound pesticides	91-110	90-99
Chlordane (6 peaks)	88-106	93-109
Toxaphene (8 peaks)	99-105	96-106
PCB-1254 (7 peaks)	93-98	102-105
PCB-1016 (8 peaks)	86-97	98-106
PCB-1260 (11 peaks)	91-102	97-112
PCB-1221 (7 peaks)	98-117	97-101
PCB-1232 (10 peaks)	89-134	92-96
PCB-1242 (9 peaks)	92-101	93-97
PCB-1248 (7 peaks)	98-101	100-103

Wastewater 2--The difficulties with this wastewater were so great that dosing and recovery experiments were not completed for several of the pesticides and PCB's. Erratic response to clean up, especially with mercury, produced inconstant background values in many instances. The greatest difficulties occurred in the first four minutes after injection. It was not unusual to experience no reduction in the interferences in undosed wastewater replicates treated with mercury and yet find substantial reductions in interferences in the dosed replicates, or vice versa (Figure A58). An impasse was reached and work with this wastewater was set aside so attention could be given to the other wastewaters. The data obtained is given in Tables A126 through A133. Recovery ranges were as follows in Table 11.

TABLE 11. RANGES OF RECOVERIES, WASTEWATER 2

	Ranges of Recoveries, %, avg	
	Zero Time	*7-Days' Storage*
Single compound pesticides	not obtained	67-158
Chlordane (6 peaks)	31-229	98-172
Toxaphene (8 peaks)	93-101	95-101
PCB-1254 (7 peaks)	93-103	89-119
PCB-1016 (8 peaks)	not obtained	not obtained
PCB-1260 (11 peaks)	90-109	91-112
PCB-1221 (7 peaks)	not obtained	not obtained
PCB-1232 (10 peaks)	not obtained	not obtained
PCB-1242 (9 peaks)	not obtained	91-128
PCB-1248 (7 peaks)	not obtained	88-161

Wastewater 3--The difficulties with this wastewater consisted of emulsion formation and erratic response to cleanup with mercury. The former was overcome sufficiently for work to proceed by techniques described in the section on wastewater analyses. The latter difficulty, although not as pronounced as with Wastewater 2, persisted throughout the work with this sample. As with Wastewater 2 the undosed replicates were more erratic as a rule than the dosed ones. As a consequence, the background "k" values were from replicates not exposed to mercury, and the dosed replicates all received cleanup with mercury. In some instances this resulted in recovery percentages which are probably too low by a few percentage points. Where study of the chromatograms and the computer printout sheets indicated in dosed samples that the background area ("k" value) in the undosed sample was not combined with the area produced by the dose, then the "k" value was not subtracted and the answers thus obtained are the "reasonable" ones. In some other instances, there is no rationale by which a reasonable answer can be produced, and these peaks should be excluded from the analysis profile of peaks. The data obtained are given in Tables A134 through A144. Aldrin, DDE, DDT and toxaphene suffered the highest losses during the seven-day storage period, with losses ranging from 12 to 17 percent. Recovery ranges were as follows in Table 12.

TABLE 12. RANGES OF RECOVERIES, WASTEWATER 3

	Ranges of Recoveries, %, avg *Zero Time*	 *7-Days' Storage*
Single compound pesticides	79-102	83-106
Chlordane (6 peaks)	78-89	98-105
Toxaphene (8 peaks)	89-99	83-88
PCB-1254 (7 peaks)	89-94	91-95
PCB-1016 (8 peaks)	92-99	92-98
PCB-1260 (11 peaks)	95-146	88-92
PCB-1221 (7 peaks)	86-97	91-93
PCB-1232 (10 peaks)	81-171	85-108
PCB-1242 (9 peaks)	79-87	109-114
PCB-1248 (7 peaks)	83-91	91-96

Wastewater 4--As mentioned earlier, Wastewater 4 was quite similar to Wastewater 3 and for this and other reasons only certain substances were dosed and recovered. This was performed at zero time, only. There were no unexpected problems met, and mercury cleanup was less erratic than with Wastewater 3 and with earlier undosed replicates of this wastewater. Treatment of an extract with BioBeads SX-2 was without beneficial effects. The data obtained are thought to be typical of that which would have been obtained with the pesticides and PCB's excluded from this test and are given in Tables A145 through A147. Recovery ranges are given in Table 13 below:

TABLE 13. RANGES OF RECOVERIES, WASTEWATER 4

	Ranges of Recoveries, %, avg *Zero Time*
Single compound pesticides	82-101
Chlordane (6 peaks)	77-97
PCB-1254 (7 peaks)	93-101

Wastewater 5--This wastewater represented a serious challenge to this program. It was an effluent from a pesticide manufacturing plant, and it contained numerous nonpriority, chlorinated pollutants which interfered with the analyses for many of the priority pollutants which are the subject of this program.

When this wastewater was being analyzed, it was found that Florisil afforded a slightly better, although inadequate, cleanup than did alumina. In another experiment, the first fraction from Florisil of a dosed sample extract was exchanged into DCM and applied to a 22mm × 31cm column of BioBeads SX-2. During elution of this column with DCM, several fractions were collected and analyzed. There was no improvement in the separation of the interfering materials from the dosed pesticides.

The ability of concentrated sulfuric acid and fuming sulfuric acid (7.5 percent) to extract interfering materials from a hexane solution of the first fraction from Florisil was tested. Concentrated sulfuric acid removed a small, but insignificant, amount of these materials, and the fuming acid was no better.

Another first fraction from Florisil, dosed with α-BHC, β-BHC, δ-BHC, heptachlor epoxide, DDE, DDD, DDT, and endosulfan sulfate, was subjected to alkaline hydrolysis using barium hydroxide. The interfering compounds were mostly destroyed as were all of the compounds of interest, except DDE. These results were to be expected when potassium or sodium hydroxides are used[11], but it had not been clear that the results would be the same when barium hydroxide was substituted.

By operating the GC on a temperature program, advantages were realized with some of the substances, for example, single compound pesticides and chlordane. This technique was applied to analyze recovered doses of γ-BHC, heptachlor, aldrin, endosulfan I, dieldrin, endrin, endosulfan II, endrin aldelhyde, and chlordane from this wastewater at zero time. The temperature was programmed from 160° to 200°C at 4° per minute. Figures A59, A60 and A61 are chromatograms of programmed injections of Florisil fractions 1, 2 and 3 from an extract of an undosed replicate. Figure A62 shows 8 single compound pesticides under programmed conditions. Figures A63, A64 and A65 are chromatograms of programmed injections of Florisil fractions 1, 2 and 3 from an extract of a dosed replicate. Scrutiny of these figures shows that determination of the quantities recovered from the dosed replicate requires great discrimination, whether the determination is based on computer integration of areas or on manually measured peak heights. Table A148 gives the recovery values from three replicates extracted at zero time. Only dieldrin defies determination completely. Recoveries for the other components ranged from 62 to 107 percent. Endrin determination gave more reasonable values under isothermal conditions than programmed conditions. Figure A66 shows the chlordane standard under programmed conditions. Figure A67 is a chromatogram of a programmed injection of Florisil fraction 1 from an extract of a replicate which had been dosed with chlordane. Peak height measurements produced the more reasonable recovery values, which are given for three dosed replicates in Table A149. Recoveries ranged from 57 to 109 percent. It is noted that the 57 percent value for peak 2, which is the heptachlor component of chlordane, checks fairly well with the 62 percent recovery obtained for heptachlor earlier.

Dosing and recovery were also carried out at zero time only with PCB-1242 and PCB-1254 combined, using the procedure in ASTM Method D3304-77. In this method, alkaline hydrolysis followed by extraction with concentrated sulfuric acid is performed to reduce non-PCB interferences. Figure A68 is a chromatogram of a Florisil fraction 1 which has been thus treated. Figure A55 is a chromatogram of an untreated Florisil fraction 1 under identical GC conditions. It may be noted that some interfering peaks remain in the treated fraction, expecially in the early part of the chromatogram. Figure A69 shows the chromatogram produced by the combined PCB standard. Figure A70 is a chromatogram of a treated Florisil fraction 1 from an extract of a dosed replicate. Three such replicates were dosed, extracted, treated, and analyzed. Table A150 gives the values obtained. The data show that peaks 1 and 5 in PCB-1242 were obscured by interferences to the extent they could not be read. Recoveries of other components ranged from 22 to 85 percent. The poor recoveries indicated by the other PCB-1242 peaks, except for peak 6, are probably the result of poor quantitation, as a consequence of interferences, rather than poor extraction. This view is supported by the fact that the indicated recoveries for PCB-1254 are much higher and uniform in values.

The other PCB's should give comparable results. Those which elute early (PCB-1016, PCB-1221, PCB-1232, PCB-1248) will be analyzed with greater difficulty and less precision and accuracy, probably, than the other late eluter, PCB-1260, in Wastewater 5.

Discussion-In Phase I, endosulfan sulfate was found to disappear almost entirely from dosed clean water replicates stored for seven days at 4° and 24°C. In Phase II, no similar inordinate disappearance of endosulfan sulfate occurred in the replicates of Wastewaters 1, 2 and 3 during storage at 4°C. The reason for this observed difference in behavior is not known. Perhaps it is related to one of the following:

(1) **The level of dosing in Phase I was only 2 percent of the level in Phase II. If the same quantity disappeared in both instances, it would not have been apparent in the Phase II work because of the differences in interferences and instrument sensitivity settings in the two phases of work.**

(2) **The oxygen demand in the wastewaters may have been so great by other substances that endosulfan sulfate did not have oxygen available for further degradation. This reasoning presumes that the degradation mode is an oxidative one. The first explanation is thought to be the more reasonable.**

In the liquid-solid chromatography study with Florisil in which doses were added to the columns in five milliliters of pure solvents, small amounts (usually 5 to 10 percent) of PCB-1260 and PCB-1232 were consistently found in the second fraction of eluate. In the dosed wastewaters, these two substances were never found in the second fraction of the eluate from Florisil. It is thought that the presence of polar impurities in the wastewaters may have been sufficient to cause all of these substances to elute completely in the first fraction.

Mercury as a cleanup agent for sulfur sometimes gave erratic results but other methods of sulfur reduction are probably no better. Under the same contract in which the work in this report was performed, another group at Southwest Research Institute investigated the procedures for analyzing for Category 10 substances in sewage sludge. This group found mercury to be superior to acid-washed copper and to tetrabutylammonium sulfite reagent[12] with respect to cleanup and with less destruction of pesticide compounds than the other two agents. For these reasons, mercury was used with the wastewaters.

Wastewater 1 was analyzed, dosed and reanalyzed with much greater ease than the other wastewaters. Wastewaters 3 and 4 gave bad, but manageable, emulsions and required reduction of interferences from sulfur. Despite the problems, the planned work with Wastewaters 3 and 4 could be conducted and satisfactory results were obtained. Wastewaters 2 and 5 presented problems which were severe enough to prevent completion of the planned experiments in some instances. Attempts to solve the problems and thus establish a method for the routine analysis of these two wastewaters were only partially successful.

ACCURACY AND PRECISION

Approach

The accuracy and precision assessment for the method was limited due to the numbers and types of analytical results obtained. In the initial design of the program three replicates of each of five wastewaters were to be analyzed for each group within this category. This was reduced, as discussed above, and for Wastewater 2 could not be successfully accomplished due to the nature of the matrix. In addition, the combining of the results of the several wastewaters was not appropriate due to the effects of levels or nature of the substances in the background or the degree of emulsion encountered upon extraction on the recovery of the dosed material. In effect, then, the data obtained can be classified as up to five sets of triplicate analyses rather than up to 15 method replicates. The conclusions of this report will be directed at general statements about the method's performance in various types of wastewater rather than a comprehensive evaluation of method performance.

For the multipeak substances (chlordane, toxaphene and the PCB's), the calculated recovery was based on an average of the peaks studied in this program. To account for the differences in concentration of the various components of these pesticides and PCB's, the recovery for each component was multiplied by the percentage of the substance it represents, as obtained from literature, and a weighted average obtained. The formula used was

$$\overline{X}_w = \left[\sum_{i=1}^{n} W_i\right]^{-1} \sum_{j=1}^{n} W_j X_j$$

where $\overline{X}_w$ = weighted average
W_i = percentage of the total compound represented by peak i
X_j = percentage recovery obtained for peak j.

Data were obtained for chlordane and for all the PCB's except PCB-1016. The peaks identified and their percentages are shown in Table 14, along wih the total percentage of the pesticide used in the evaluation. The percentages were obtained from reference material showing representative chromatograms, and these chromatograms were matched with those generated on this program. In some cases, the reference chromatograms were obtained using a different column, and not all studied peaks could be identified with respect to the relative contribution to the compound. In the case of toxaphene and PCB-1016, no reference material was found that indicated the percentage composition, and a simple average was calculated for the peaks under study. This procedure gave acceptable results, and had the advantage of smoothing the fluctuations in the data for those peaks which represented small absolute amounts close to the detection limit for that compound.

TABLE 14. RELATIVE COMPOSITION OF MULTI-PEAK SUBSTANCES
(PERCENT OF TOTAL WEIGHT)

Substance	*Peak Number* *1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*Total*
Chlordane	—*	7	13	19	24	—						63
AR 1254	13.2	17.3	13.6	15	—	—	—	—				59.1
AR 1260	2.7	4.7	—	—	—	—	—	—	9.8	11.0	4.2	32.4
AR 1242	11.3	11	11.5	—	—	8.8	6.8	10.3	3.6			63.3
AR 1248	5.2	8.3	15.6	9.7	9.3	19.0	6.6					73.7
AR 1232	16.2	—	17.8	9.6	6.8	—	—	4.2	3.4	4.6		62.6
AR 1221	31.8	19.3	10.1	[20.8]⁺		5.4	1.7					89.1

* No information obtained

+ Combined total

Accuracy of a method refers to its ability to obtain the true value average, within the limits of the variability inherent in the method. For this study, the accuracy standard used is the percent recovery of the amount of the pesticide or PCB dosed into the wastewater, with 100 percent representing an accurate method. Precision relates to the inherent variability in the analytical data, irrespective of the mean level. Thus, a method can be precise, meaning small variability among measurements, without being accurate. For this study, the precision measurement used was the range, or the difference between the highest and lowest recoveries. While the range is sensitive to single extreme values, it was deemed more appropriate for these data than other measures, given the number of observations available.

The recoveries obtained after 7 days storage at 4°C were examined to determine the effects of storage on the observed levels of these pesticides and PCB's. These recoveries are based on the total amount present at time zero, not on the dosed amount, since it is assumed any degradation would affect both the initial amount and the dosed amount in equal proportions. The variability among these triplicate determinations, however, can be combined with those of the 0-day analyses to evaluate the precision of the method.

In order to provide some composite viewpoint on the results, three combinations of the groups in this category have been established; single compound pesticides, multiple compound pesticides, and PCB's. The rationale behind this is that the method could be expected to perform similarly within these groups, and it allows a more general statement of the applicability of the method.

Results

Wastewater 1--

As described above, Wastewater 1 was a relatively clean effluent, and good recoveries of the dosed amount were generally obtained. Considering the single-compound pesticides as a class, average recoveries from 91 percent for α-BHC to 110 percent for endosulfan II were obtained, and over 50 percent of the average recoveries fell between 95 and 99 percent. The preservation recoveries were very good, ranging from 90 percent for endosulfan II to 99 percent for α-BHC. The background levels on Wastewater 1 were small, which means that the level at which the analyses were run was the level of the dose, ranging from 1 to 15 parts per billion. The conclusion is that for this type of wastewater, single-compound pesticides can be analyzed with greater than 90 percent expected recovery and that the pollutants will persist on storage at 4°C.

Using the range of observed values, the method demonstrated good precision on both the method and the preservation results for this wastewater. These ranges were combined for determination of method precision since all represent the ability of the method to produce repeatable results. The average range observed was 5.5, with 70 percent of the values falling at or below a range of 6 from highest to lowest percent recovery. As a result, the method can be said to exhibit good precision with respect to the analysis of single-compound pesticides in this wastewater.

Similar results were obtained from the analyses of the multicompound pesticides in Wastewater 1. Using the combined recoveries for chlordane, based on peaks 2 through 5, an average recovery of 94 percent was observed, and for toxaphene an average of 103 percent was recovered. These fall within the span of recoveries obtained for the single-compound pesticides and the results can be considered equivalent. The amount present after 7 days storage at 4°C was 99 percent for chlordane and 101 percent for toxaphene, again indicating no problem exists in storing samples for these compounds in this wastewater.

The precision of these analyses was also good, with a range of 2-3 percent recovery on all four sets of replicates. As discussed above, the averaging (weighted for chlordane) of the recoveries has a smoothing effect on the observed recoveries, and it might be expected that this would provide a small range of observed concentrations.

The weighted average recoveries for the PCB's in Wastewater 1 are shown in Table 15 for the seven PCB's under study, along with the range of values observed in each replicate set. The lowest observed recovery was 92 percent for PCB-1016 (based on a simple average recovery for all peaks) and the high was 104 percent for PCB-1221. Again, these lie within the range observed for the pesticides and indicate that for this wastewater equivalent results can be expected for any of the pesticides or PCB's. The amount present after 7 days cold storage went from a minimum of 94 percent for PCB-1232 and PCB-1242 to a maximum of 108 for PCB-1260.

TABLE 15. SUMMARY OF PCB'S IN WASTEWATER 1

	Method Recovery		*Preservation Recovery*	
PCB	***Weighted Avg.***	***Range***	***Weighted Avg.***	***Range***
1254	95	6	104	7
1016	92	4	101	5
1260	94	3	108	5
1221	104	6	100	11
1232	97	7	94	4
1242	98	4	94	1
1248	99	0	101	7

The precision of these determinations at the levels studied was good, with only one range, that for the preserved PCB-1221, exceeding seven units. The median range was 6 percent recovery, which corresponds favorably to the values obtained above.

In summary, for this relatively clean effluent, the method performed well for all pesticides and PCB's studied, with recoveries greater than 90 percent and an average range of 5 to 6 percent recovery encompassing the triplicate determinations. No significant effect upon the results was noted after storage for 7 days at 4°C.

Wastewater 2--

The interferences present in Wastewater 2 presented great difficulty in obtaining the necessary data for the accuracy and precision evaluation. There was a lack of a consistent background for comparing the dosed samples to the undosed, and there were difficulties in quantifying the substances of interest due to the high background. For the single-compound pesticides there were no recoveries calculable for the 0-day analyses and only preservation and precision data were available. The recoveries on preservation ranged from a low of 67 percent for heptachlor to a high of 158 for endrin aldehyde, and evidenced the problem discussed above in obtaining good quantitation of the studied compounds. Similarly, the range for the three determinations of each pesticide went from a low of 3 percent recovery to a high of 105. Thus, for this wastewater no satisfactory accuracy or precision could be expected for the single-compound pesticides.

The multicompound pesticides displayed a somewhat better pattern. For chlordane a recovery of 98 percent was calculated for the initial analysis of the dosed sample. However, this was based on peaks 4 and 5 only, 43 percent of the composition of chlordane, to avoid difficulties with interferences and quantitation of peaks 2 and 3. The preservation recovery was 98 percent, and indicated no problem with storage at 4°C for this pesticide. Toxaphene recovery was similar, with 96 percent at the 0-day and 99 percent at the 7-day. For these pesticides good accuracy was obtained in this wastewater.

The precision of these determinations was not as good as for the previous wastewater, but could be considered adequate. The worst case was for chlordane at 0-day, with a range of 15 percent, and the best for chlordane at 7 days with a range of only 2 percent. The range for the toxaphene was 9 percent in both instances.

For the PCB's, the summary data are shown in Table 16. Only two, PCB-1254 and PCB-1260, could be adequately quantitated at the initial analyses. These two are relatively late eluters and thus avoid many of the difficulties which arise in the first four minutes of the chromatogram. An average recovery of 100 percent was observed for PCB-1254 and an average of 94 percent for PCB-1260. The preservation study gave valid recoveries for only PCB-1242 and PCB-1248 in addition to the two above, and the recoveries went from a low of 98 percent for PCB-1254 and PCB-1248 to 108 percent for PCB-1242. The preservation recovery for PCB-1260 was 102 percent. The ranges of the replicate determinations went 0 for PCB-1248 to 17 for PCB-1260, both on preservation recoveries, with the balance falling between 6 and 10 percent recovery.

TABLE 16. SUMMARY OF PCB'S IN WASTEWATER 2

	Method Recovery		*Preservation Recovery*	
PCB	***Weighted Avg.***	***Range***	***Weighted Avg.***	***Range***
1254	100	7	98	6
1016	—	—	—	—
1260	94	10	102	17
1221	—	—	—	—
1232	—	—	—	—
1242	—	—	108	8
1248	—	—	98	0

The overall conclusions for this wastewater is that quantification problems arising from the background and only partially successful cleanup prevent the accurate determination of the recovery of the dosed amounts of most of the pesticides and PCB's. The quantification problems also result in a lack of consistently good precision among determinations, and the method did not perform satisfactorily in this medium.

Wastewater 3--

Wastewater 3 was another relatively clean wastewater, but there were problems with obtaining consistent background values and with emulsions. For the single-compound pesticides, the method recoveries went from a low of 79 percent for α-BHC to a high of 102 percent for endosulfan sulfate. In general, lower recoveries were obtained from this wastewater than from Wastewater 1, with only slightly more than half of the compounds being recovered at greater than 90 percent. These are two possible reasons for this. First, since the background was erratic with the mercury cleanup, the background values used were those from samples not cleaned up, and the background used in the calculations may be higher than the actual background present after cleanup. Second, the greater degree of emulsion experienced with this wastewater can lead to less complete recovery of the extracting solvent and hence of the desired material. In site of this, adequate recovery was obtained on all these pesticides.

The preservation recoveries were good for these compounds, with a span from 83 percent for Aldrin to 106 percent for γ-BHC. Twelve of the 16 recoveries were in excess of 90 percent upon seven days storage at 4°C, which indicates no problem in holding samples in wastewater of this type. The triplicate analyses gave good precision for both sets of samples, and all but one of the calculated ranges fell at or below seven percent. The mean range was 3.9 percent and indicated good repeatability among the measurements of the single-compound pesticides for this wastewater.

The results for the two multicompound pesticides were similar to the single-compound results. The recovery of chlordane was 87 percent with a range of 4, and of toxaphene was

93 percent with a range of 10. In both cases the recovery was lower than the corresponding value in Wastewater 1, but must certainly be considered acceptable results. The preservation recoveries were 101 percent for chlordane but only 86 percent for toxaphene, with ranges of 4 and 3, respectively. The toxaphene recovery was adequate, but not as good as in Wastewater 1, while the chlordane results were essentially the same as in Wastewater 1. The precision obtained was similar to that for the single-compound pesticides.

The PCB data followed the pattern described above with good method recoveries, although lower than on the first wastewater, with values from 82 percent for PCB-1242 to 106 percent for PCB-1221. The summary of results is shown in Table 17. The preservation recoveries observed were from 92-95 percent with the exception of PCB-1242, which showed 112 percent recovery. However, this represents only 92 percent of the original dosed amount, and may indicate that the 0-day value was inordinately low. The precision was generally good, with ranges of 5 percent or less on all but two samples, 8 on PCB-1232 on the initial analyses and 17 on PCB-1221 at 7 days.

TABLE 17. SUMMARY OF PCB'S IN WASTEWATER 3

	Method Recovery		*Preservation Recovery*	
PCB	*Weighted Avg.*	*Range*	*Weighted Avg.*	*Range*
1254	91	0	95	5
1016	95	5	94	1
1260	90	3	92	1
1221	106	5	92	17
1232	89	8	93	3
1242	82	3	112	4
1248	86	2	93	1

The general conclusion is that the method performs well in this matrix on all pesticides and PCB's studied. It may be possible to obtain recoveries that are better than those shown here, by stabilizing the background level and/or by determining solvent recovery and adjusting the calculated concentration, but without this extra burden the results are satisfactory to good. Good repeatability was obtained for all three classes of compounds, generally on concentrations in the low parts per billion range of concentration.

Wastewater 4--

The fourth wastewater did not present any major difficulties upon analysis and the results confirm that. For the single-compound pesticides the method recoveries went from a low of 82 percent for α-BHC to a high of 101 percent, with all but two showing recoveries of 93 percent or greater. The precision was very good, with ranges from 0 to 5 percent and a median range of 3 percent. The recovery of chlordane was 92 percent with a range of 5 percent and of PCB-1254 was 99 percent with a range of 4 percent. Thus, the method worked well for these compounds both with respect to accuracy and precision.

Wastewater 5--

The fifth wastewater presented many problems on analysis. There was a high background level and this presented problems upon analyzing for the pesticides of interest. The recoveries of the dosed amount went from 62 percent for heptachlor to 307 percent for dieldrin. It should be noted, however, that the dieldrin spike was extremely small relative to the background and

that such results are not uncommon when this is the case. The background, as with Wastewater 2, causes poor quantitation of the compounds and hence less precise results as well. High values >20 were observed for the range of determinations for endrin, dieldrin and endrin aldehyde, the latter two where the spike-to-background ratio was extremely low.

Chlordane was recovered at 93 percent, with a range of 3 percent, using temperature programming on the GC, and this variation seemed to provide good results for this pesticide. The Aroclors studied were combined for purposes of determining how well they could be chromatographed together, for purposes of the follow-on method study, and the quantitation discussed earlier could not be followed exactly. Of the numbered peaks used for quantitation, based upon the data presented above, only peaks 2, 3, 6 and 7 of PCB-1242 were separable from peaks of PCB-1254, and only peaks 3 and 4 of the latter could be used. These give recoveries of 53 percent with a range of 21 and 82 percent with a range of 6. Obviously, better results were obtained with the higher-eluting PCB where the background was considerably lower and quantitation was easier.

SUMMARY OF PHASE II

Fully activated Florisil PR and alumina mixed with 10 percent by weight water were put in glass columns, dosed with pesticides the 25 pesticides and PCB's in hexane, and eluted with hexane or mixtures of hexane and ethyl ether to determine their separation patterns and recoveries. Each dosing and eluting procedure was done five times. Recoveries were excellent for all substances from both columns. However, the separation patterns are different, with the distributions of substances in the fractions from the Florisil PR being preferred.

Five wastewaters were analyzed in triplicate for each of the 25 substances. All of the wastewaters contained interferences that presented mild to severe effects on the analyses that were performed by computer-assisted gas chromatography. The interferences consisted of elemental sulfur, chlorinated compounds other than those of interest, and various other electron capture sensitive materials. Wastewaters 2 and 5 contained especially troublesome materials of this nature. Florisil provided better cleanup than did the alumina, although the margin of difference was usually small. Mercury was used to remove sulfur interference, but it performed erratically. Wastewaters 3 and 4 produced emulsions whether the extracting solvent was 15 percent DCM in hexane or pure DCM. Work with Wastewater 1 was relatively trouble free. Wastewater 2 contained no pollutants of interest. The other wastewaters contained (tentatively) one to three of the pollutants of interest, all at levels below 10 parts per billion except for endrin in Wastewater 5 at 32 parts per billion.

The five wastewaters were dosed with the pesticides and PCB's and analyzed to provide data for accuracy and precision assessments. Three of the dosed samples were analyzed immediately, and three were analyzed after storage for 7 days at 4°C. The schedule of dosing and analyzing was completed for Wastewaters 1 and 3. With Wastewater 4, the number of samples dosed and analyzed was reduced to provide additional time for method development. The problems with Wastewaters 2 and 5 were never completely overcome and the schedules were only partially completed.

Accuracy and precision were satisfactory to very good for Wastewaters 1, 3 and 4 but ranged from unsatisfactory to good for those substances which could be analyzed in Wastewaters 2 and 5. There was no indication that extraction from the wastewaters or recovery from the cleanup columns were responsible where less than good results were obtained. Rather inadequate cleanup resulted in a background of interferences which were often too high or too variable for good quantitation. In Wastewater 2, these interferences were primarily nonchlorinated materials which, nevertheless, produced responses from the electron capture detector. In the case of Wastewater 5, the interferences are considered more serious in nature, since they consisted of chlorinated substances produced by a pesticide plant, but they were primarily compounds not of concern in this program.

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APPENDIX A

GAS CHROMATOGRAPHY FIGURES

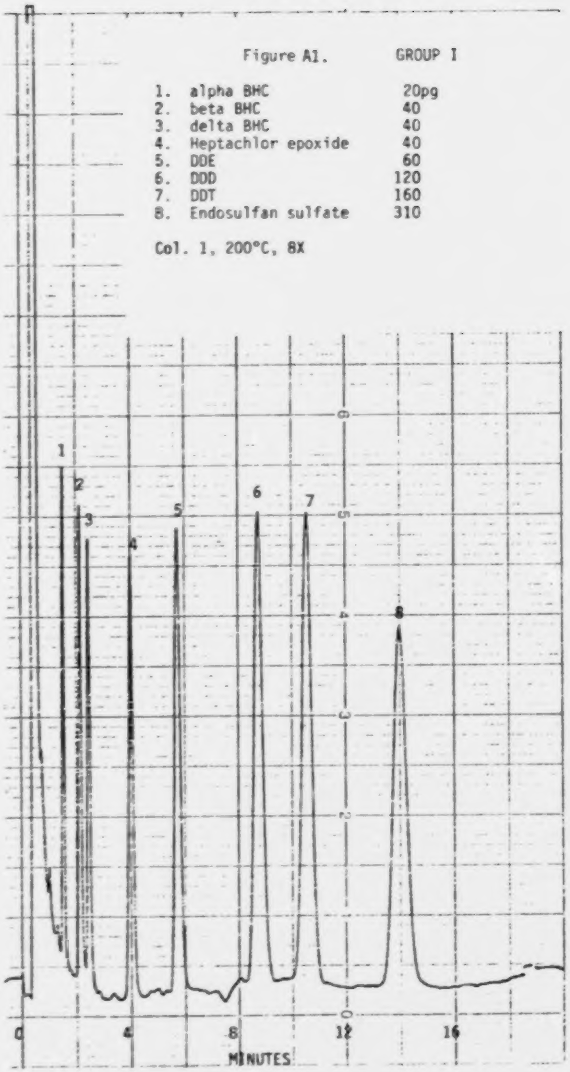


Figure A1. GROUP I

1.	alpha BHC	20pg
2.	beta BHC	40
3.	delta BHC	40
4.	Heptachlor epoxide	40
5.	DDE	60
6.	DDD	120
7.	DDT	160
8.	Endosulfan sulfate	310

Col. 1, 200°C, 8X

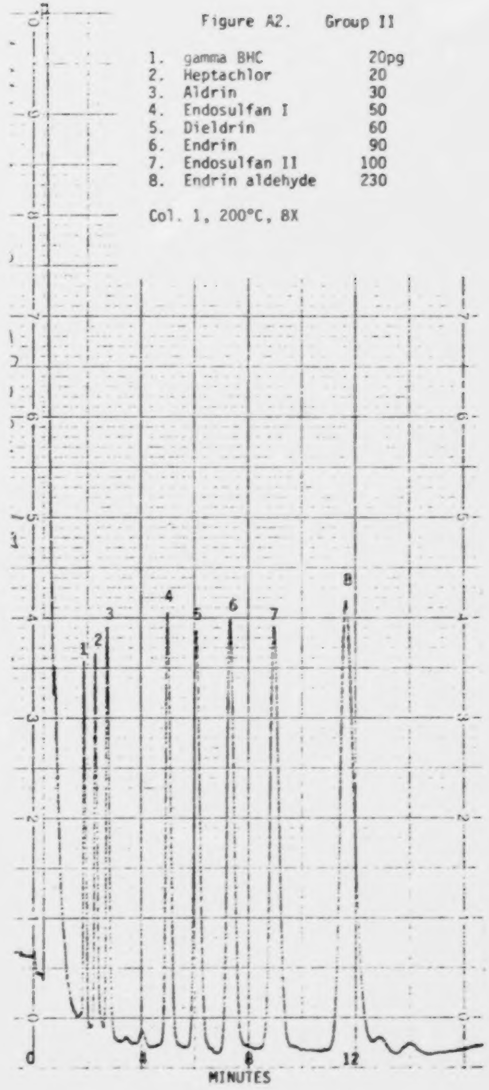


Figure A2. Group II

1.	gamma BHC	20pg
2.	Heptachlor	20
3.	Aldrin	30
4.	Endosulfan I	50
5.	Dieldrin	60
6.	Endrin	90
7.	Endosulfan II	100
8.	Endrin aldehyde	230

Col. 1, 200°C, 8X

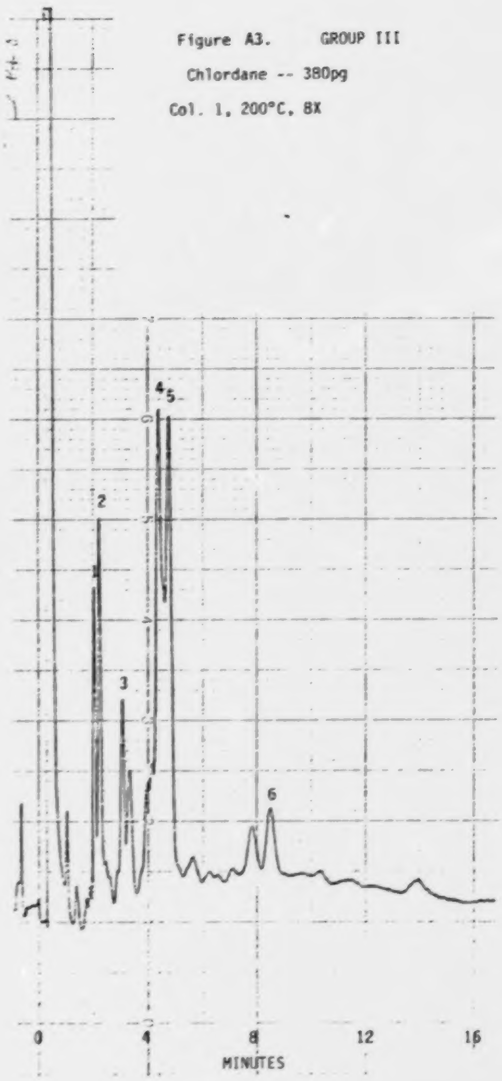


Figure A3. GROUP III

Chlordane -- 380pg

Col. 1, 200°C, 8X

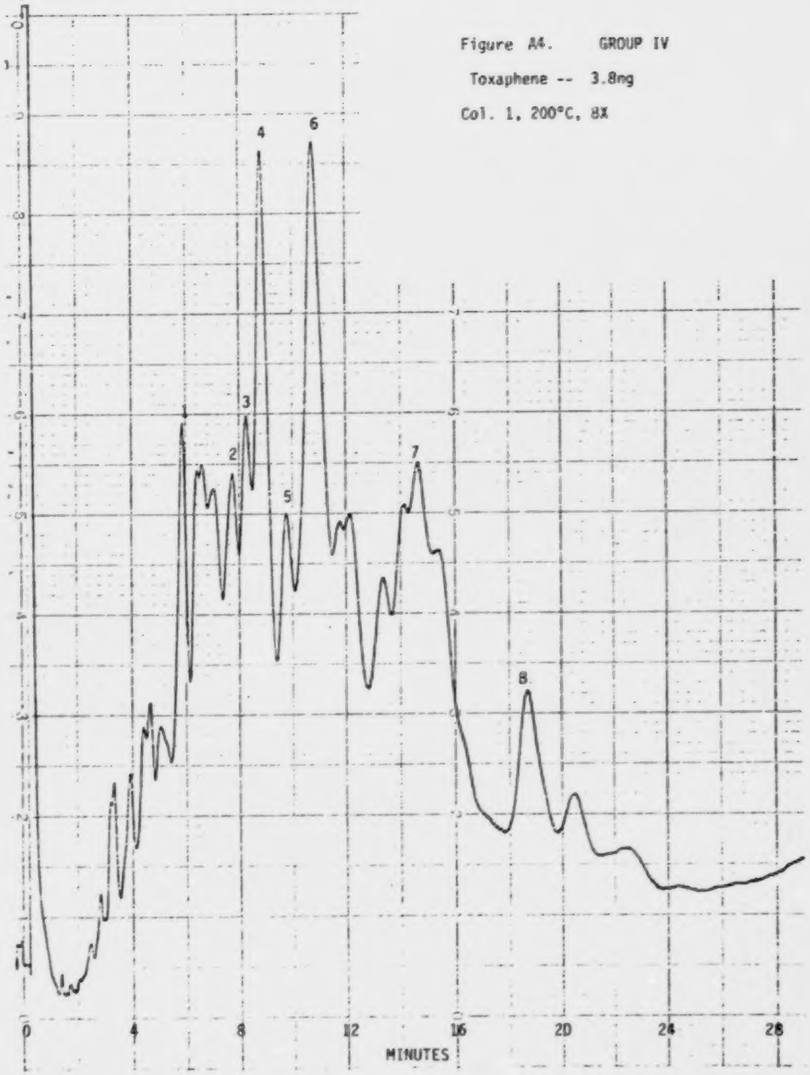


Figure A4. GROUP IV

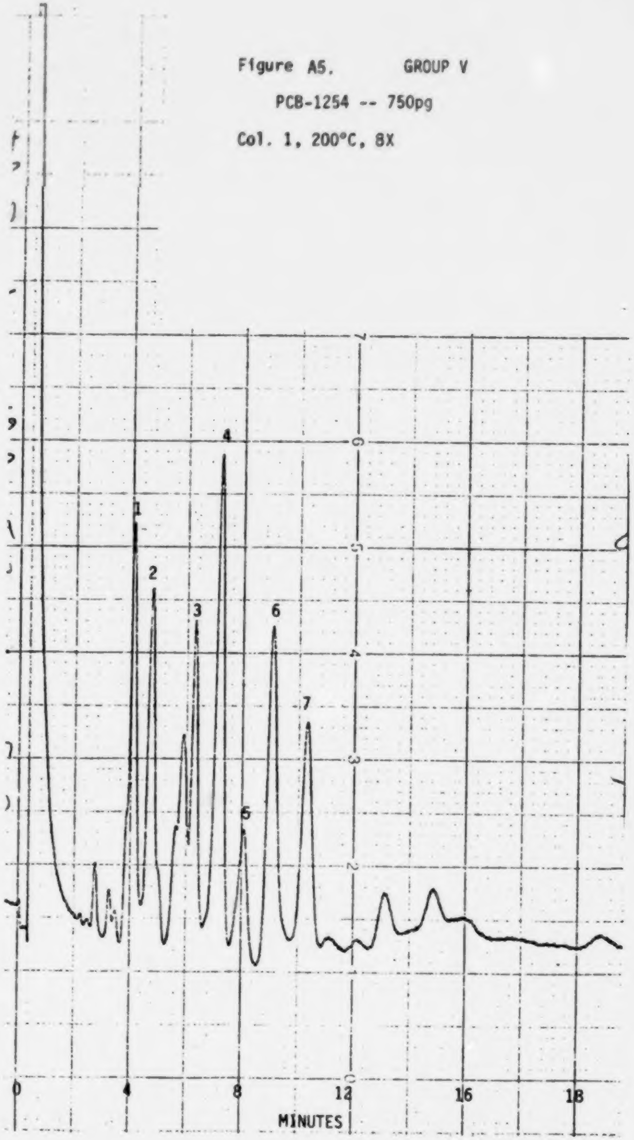
Toxaphene -- 3.8ng

Col. 1, 200°C, 8X

Figure A5. GROUP V

PCB-1254 -- 750pg

Col. 1, 200°C, 8X



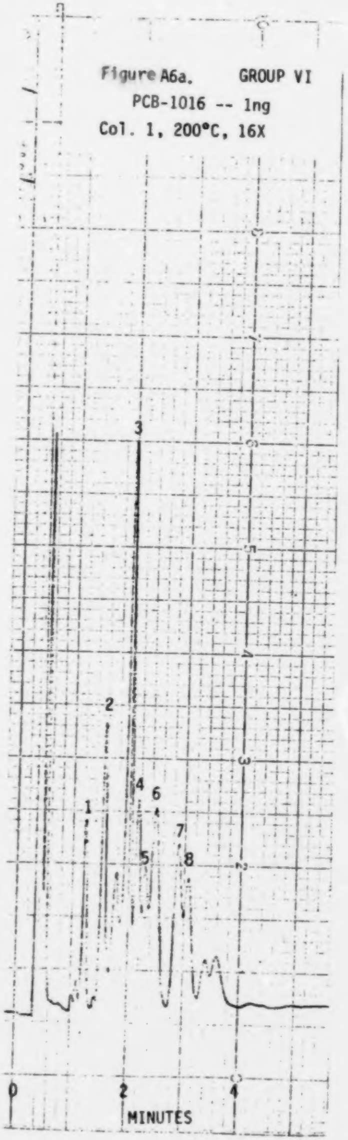


Figure A6a. GROUP VI
PCB-1016 -- 1ng
Col. 1, 200°C, 16X

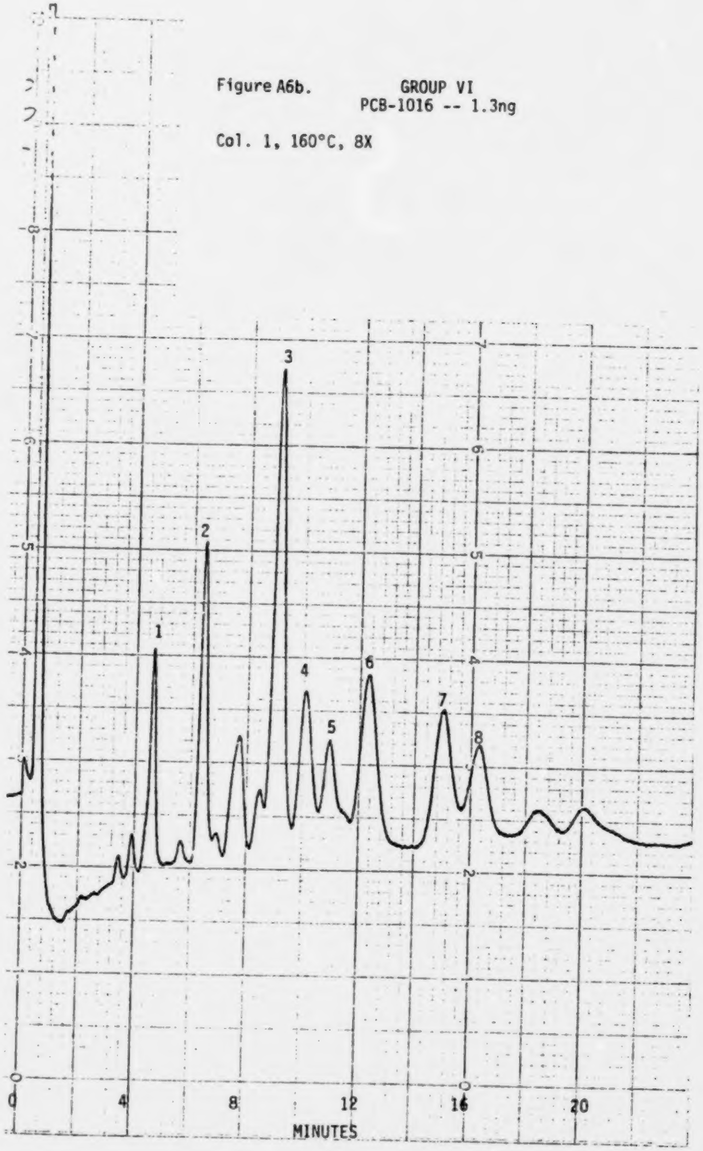
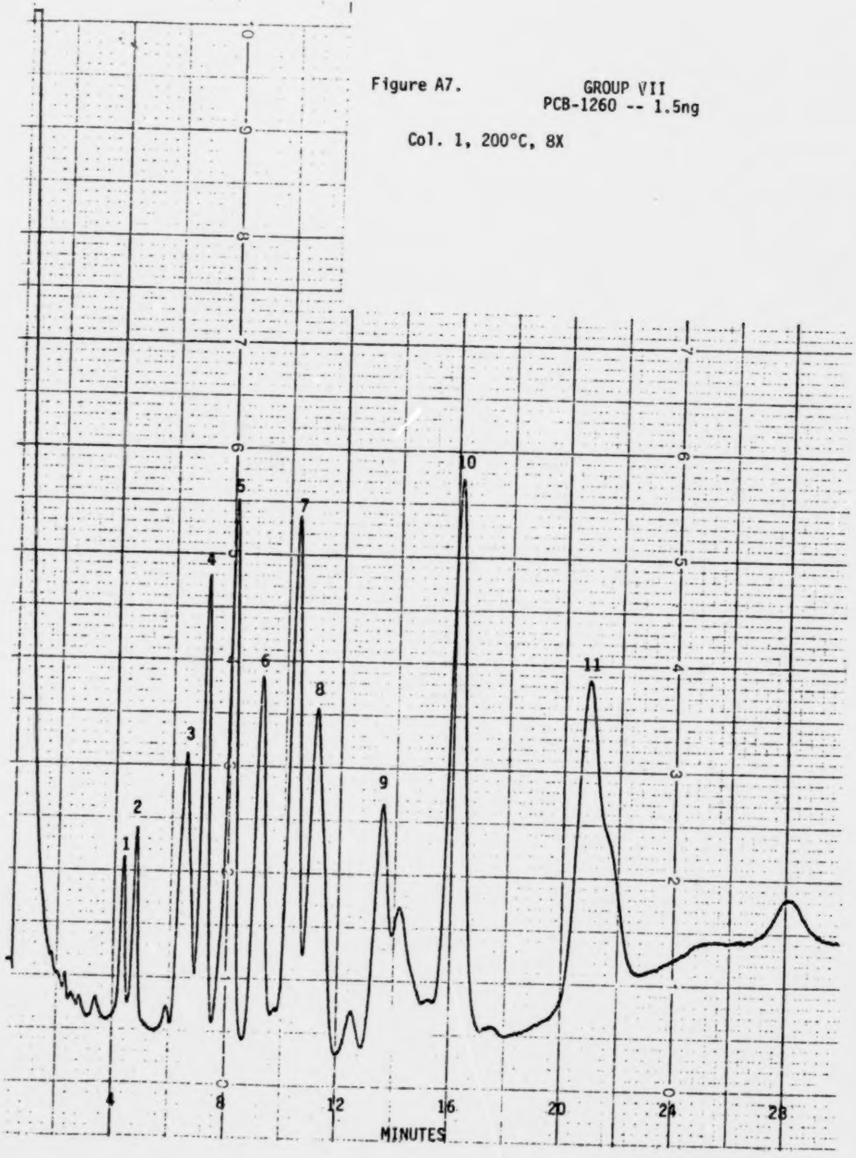


Figure A6b. GROUP VI
PCB-1016 -- 1.3ng
Col. 1, 160°C, 8X

Figure A7. GROUP VII
PCB-1260 -- 1.5ng

Col. 1, 200°C, 8X



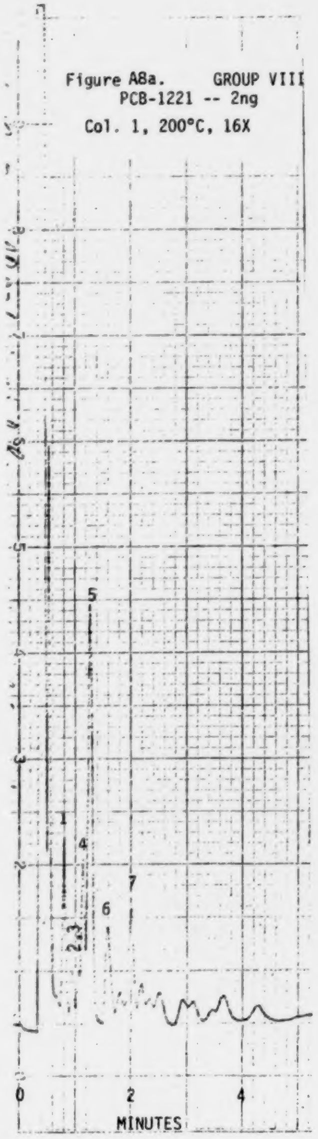


Figure A8a. GROUP VIII
PCB-1221 -- 2ng
Col. 1, 200°C, 16X

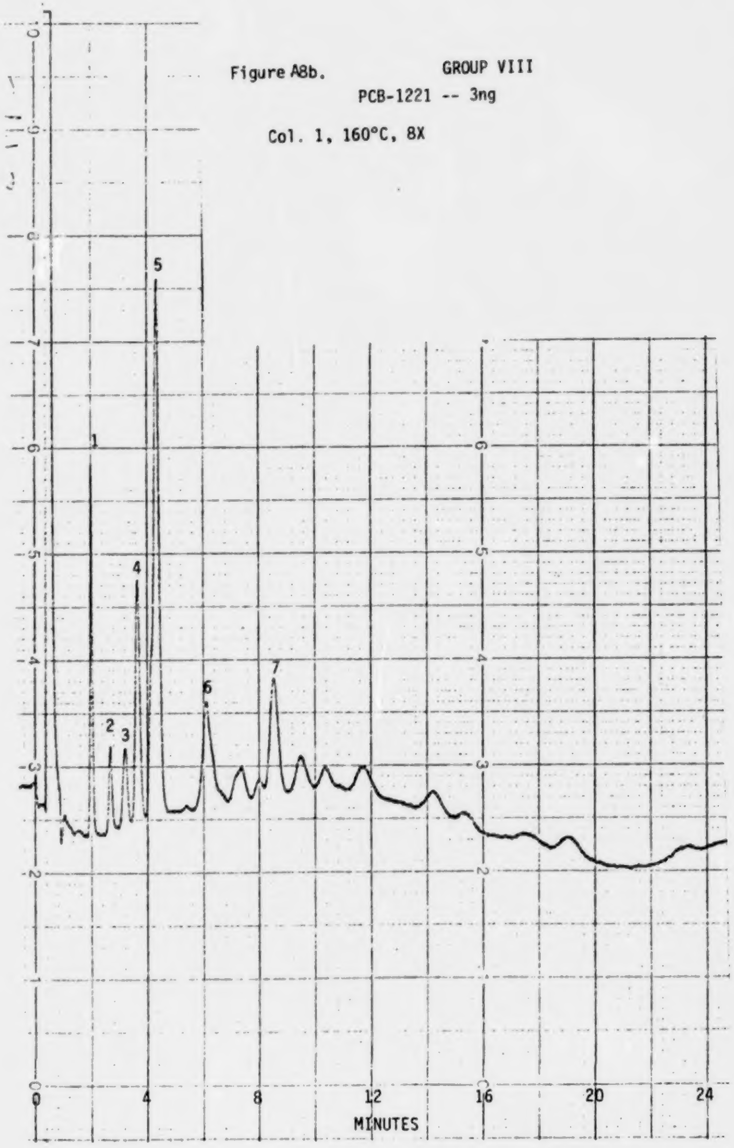


Figure A8b. GROUP VIII
PCB-1221 -- 3ng
Col. 1, 160°C, 8X

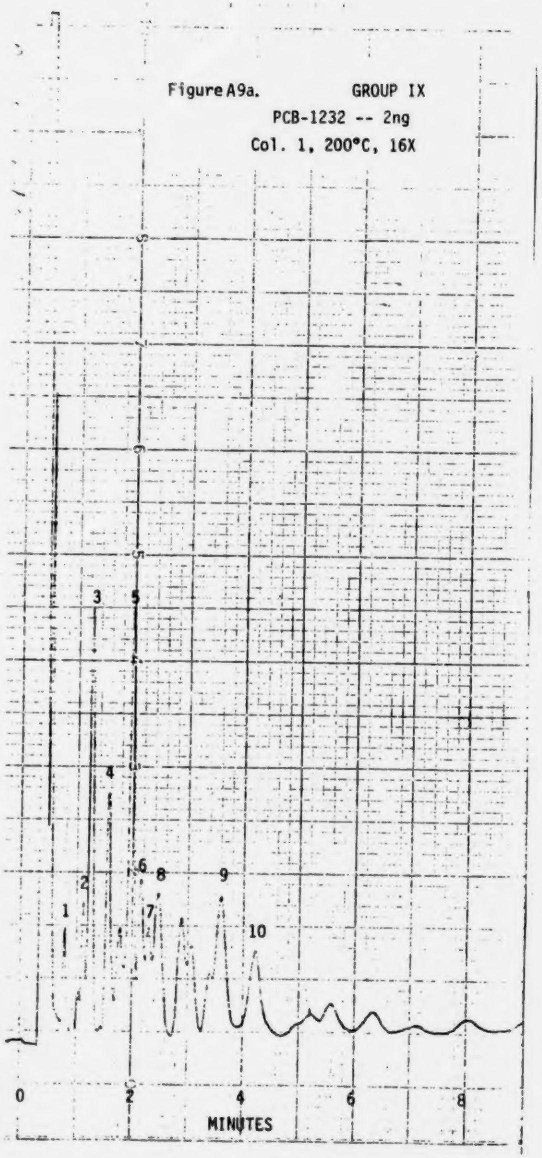


Figure A9a. GROUP IX
PCB-1232 -- 2ng
Col. 1, 200°C, 16X

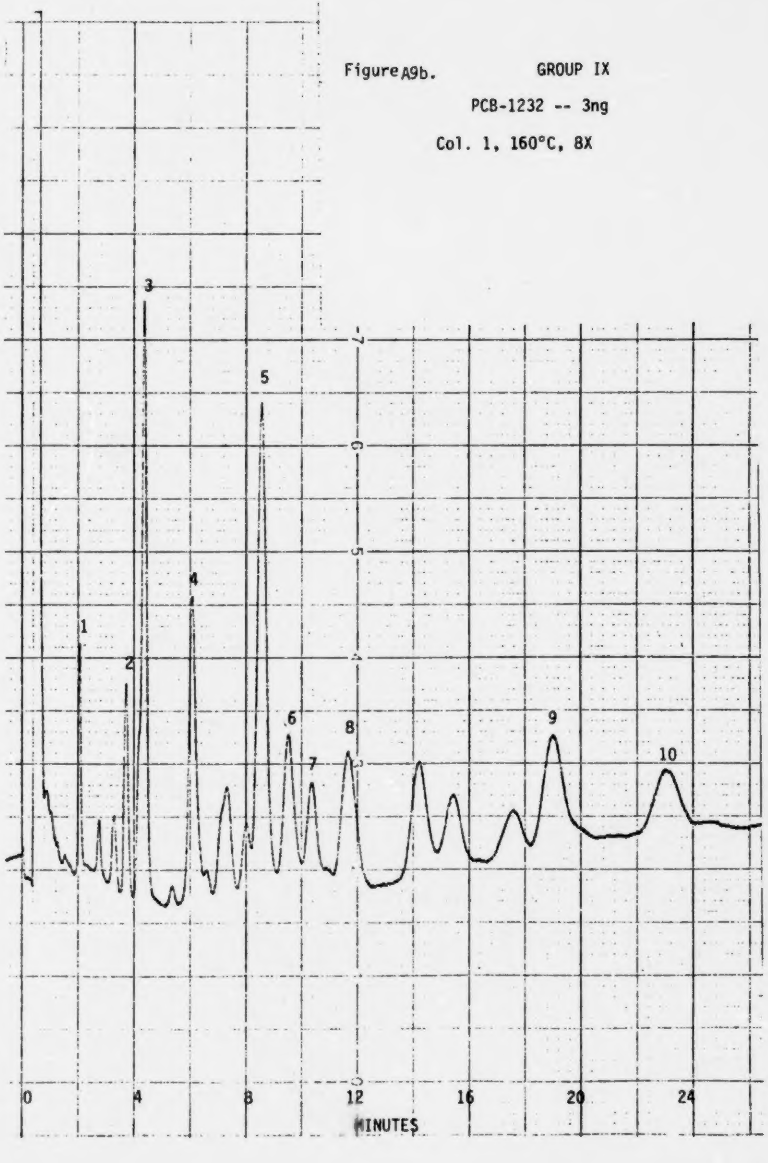


Figure A9b. GROUP IX
PCB-1232 -- 3ng
Col. 1, 160°C, 8X

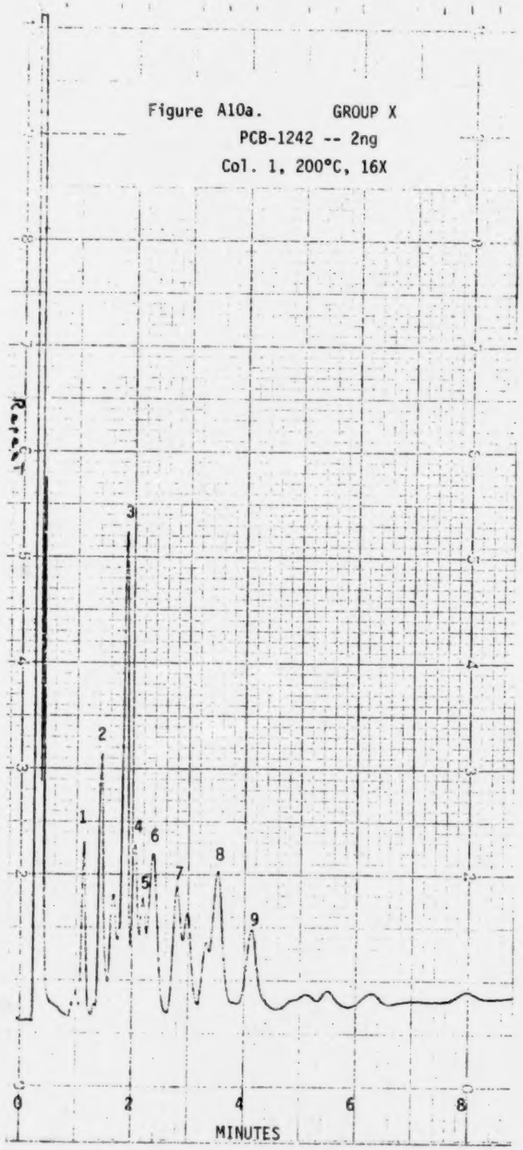


Figure A10a. GROUP X
PCB-1242 -- 2ng
Col. 1, 200°C, 16X

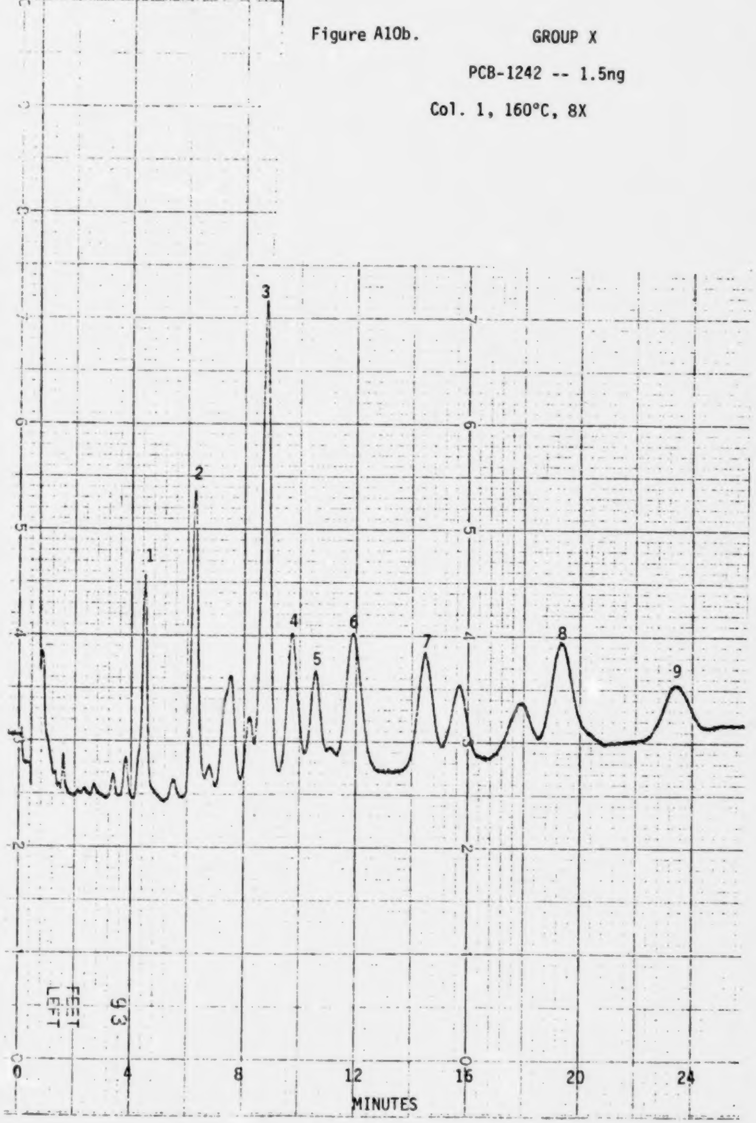


Figure A10b. GROUP X
PCB-1242 -- 1.5ng
Col. 1, 160°C, 8X

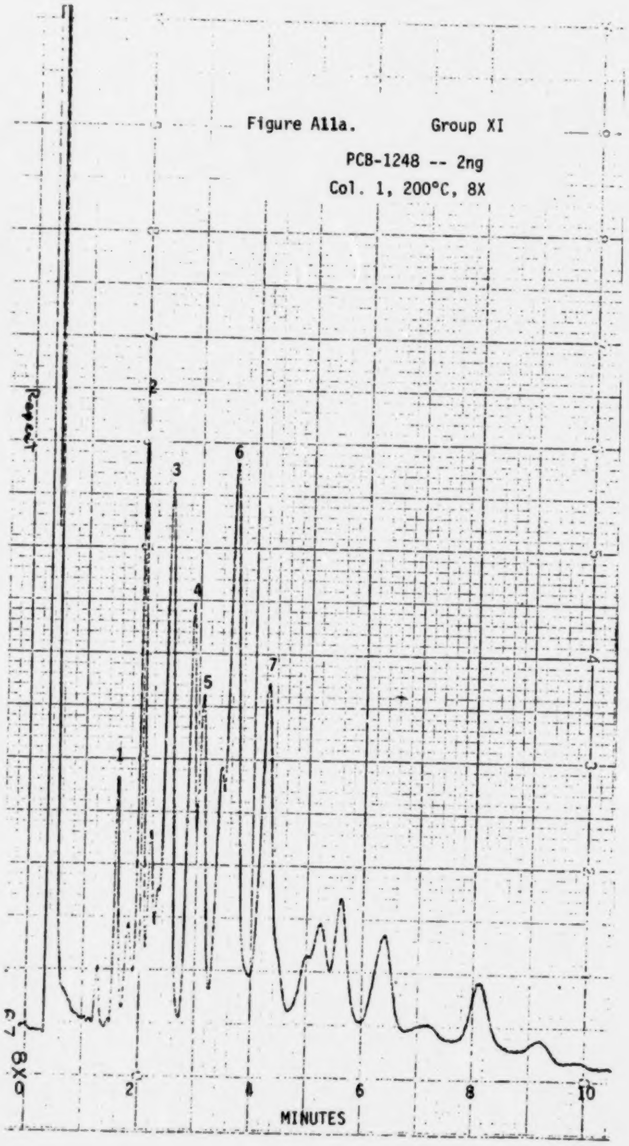


Figure A11a. Group XI

PCB-1248 -- 2ng

Col. 1, 200°C, 8X

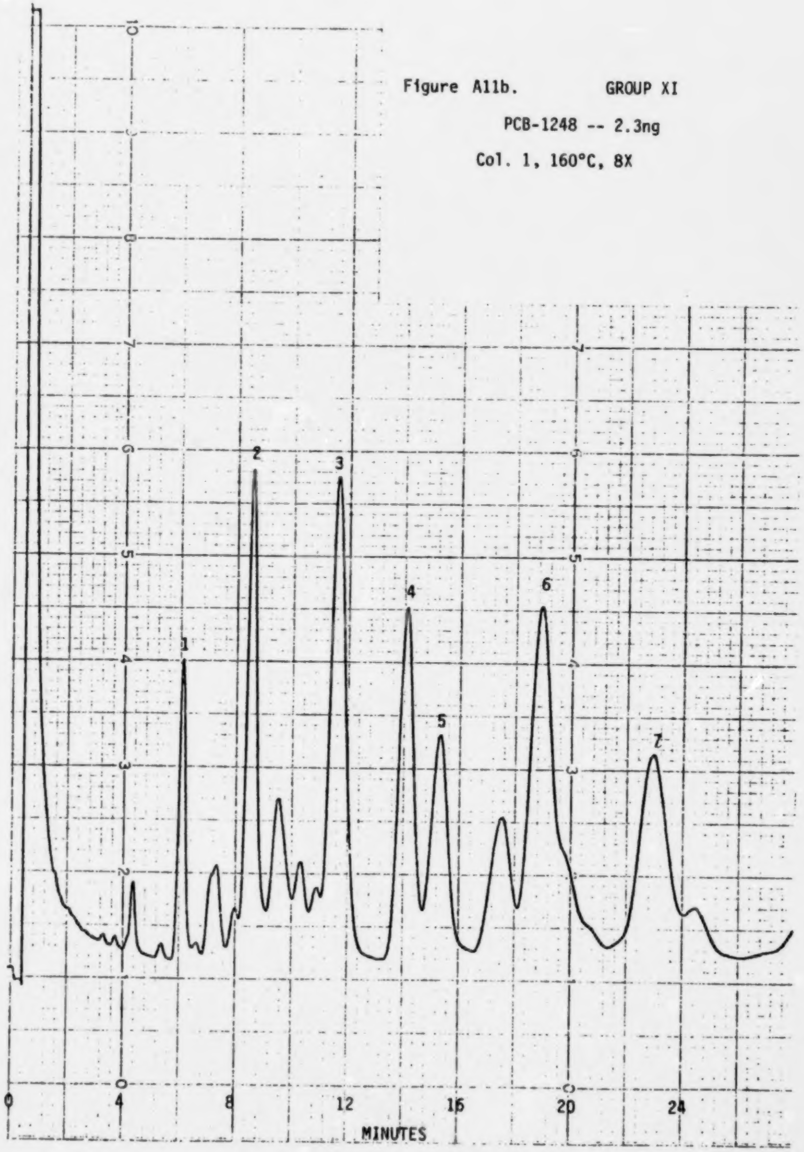


Figure A11b. GROUP XI

PCB-1248 -- 2.3ng

Col. 1, 160°C, 8X

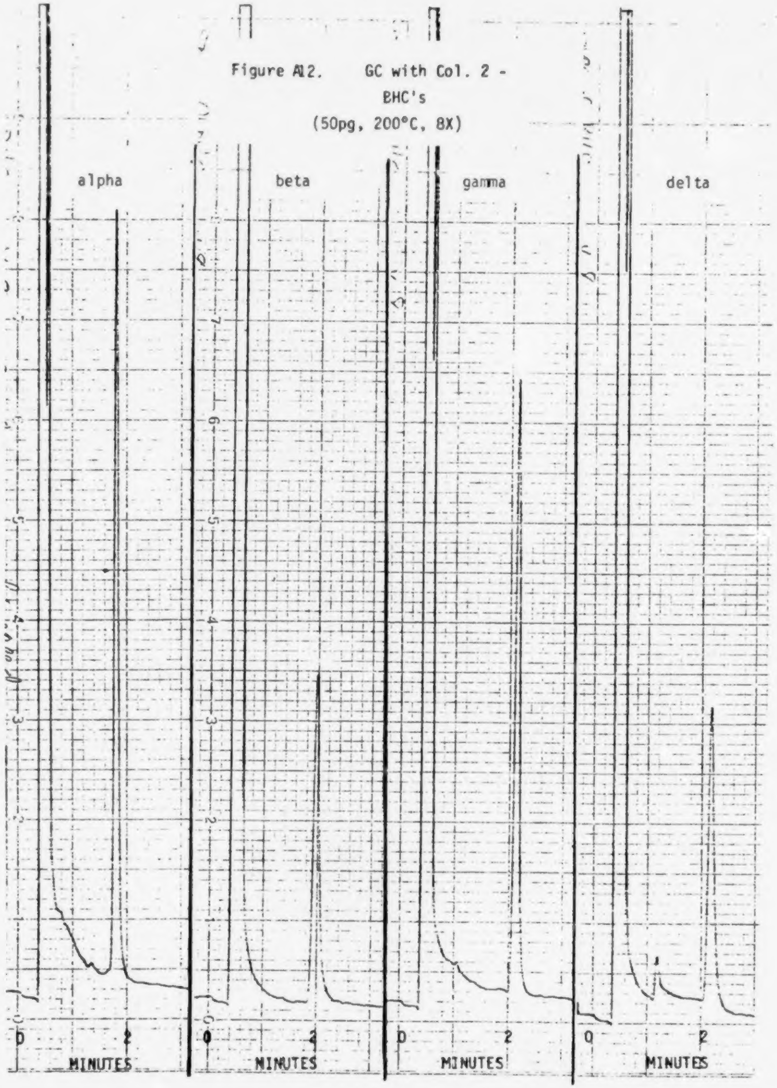


Figure A12. GC with Col. 2 - BHC's (50pg, 200°C, 8X)

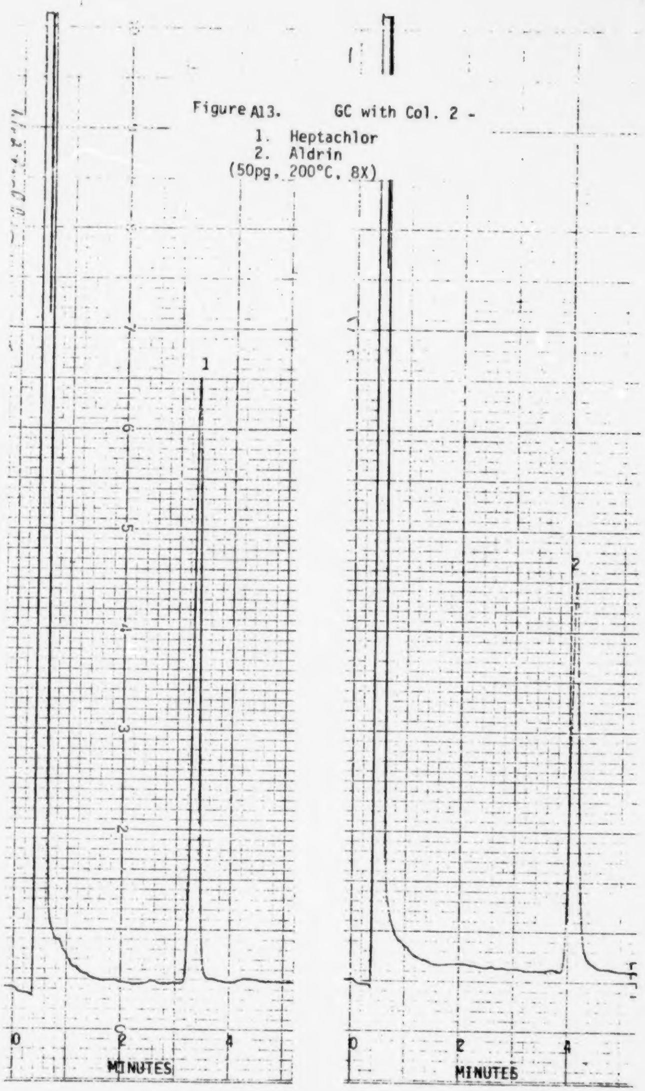


Figure A13. GC with Col. 2 -
1. Heptachlor
2. Aldrin
(50pg, 200°C, 8X)

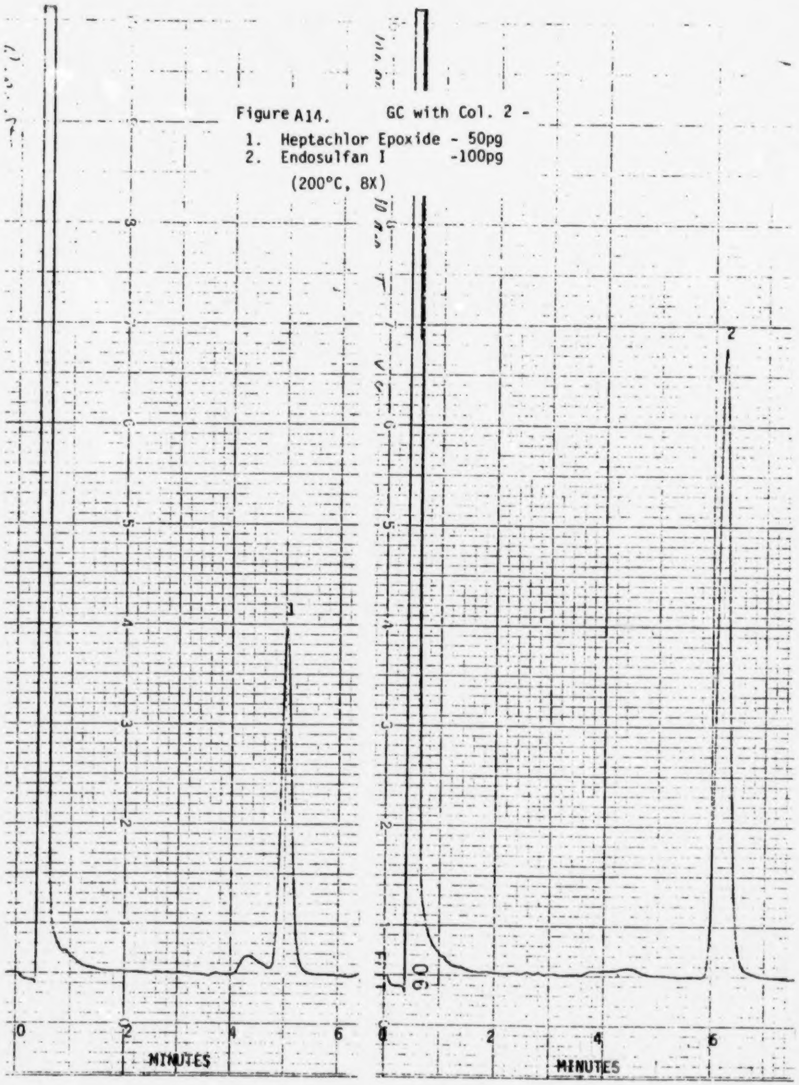


Figure A14. GC with Col. 2 -
1. Heptachlor Epoxide - 50pg
2. Endosulfan I -100pg
(200°C, 8X)

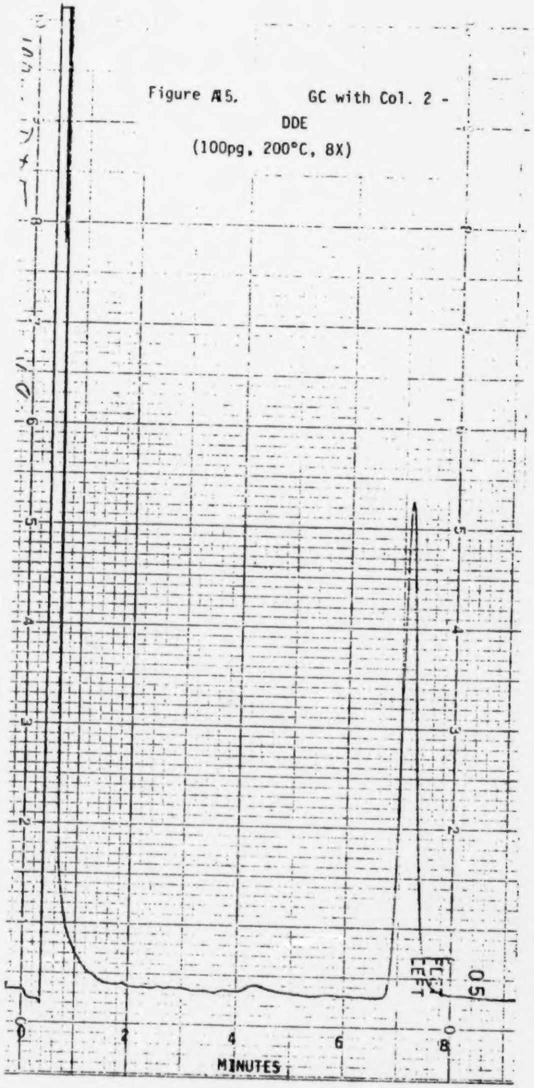


Figure A15. GC with Col. 2 -
DDE
(100pg, 200°C, 8X)

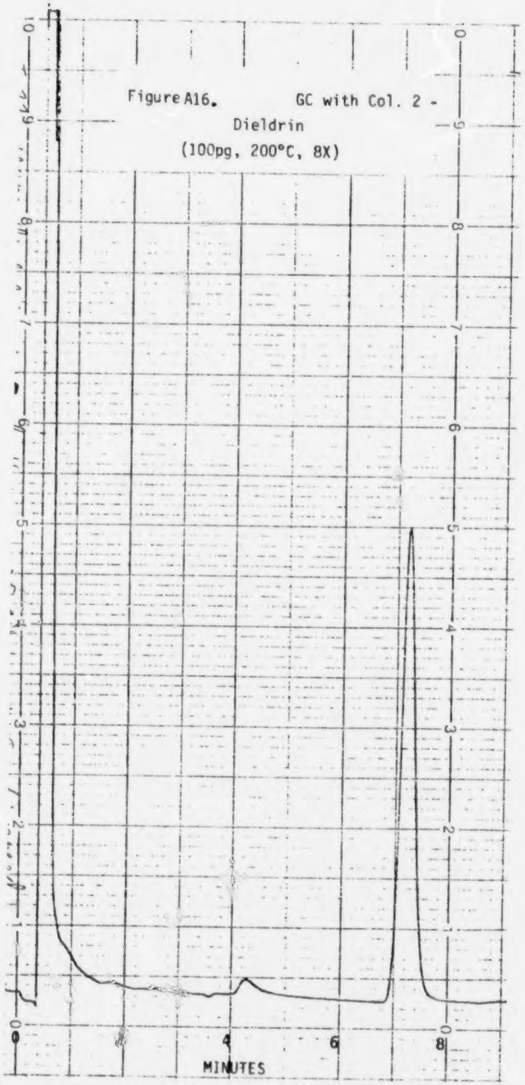


Figure A16. GC with Col. 2 - Dieldrin (100pg, 200°C, 8X)

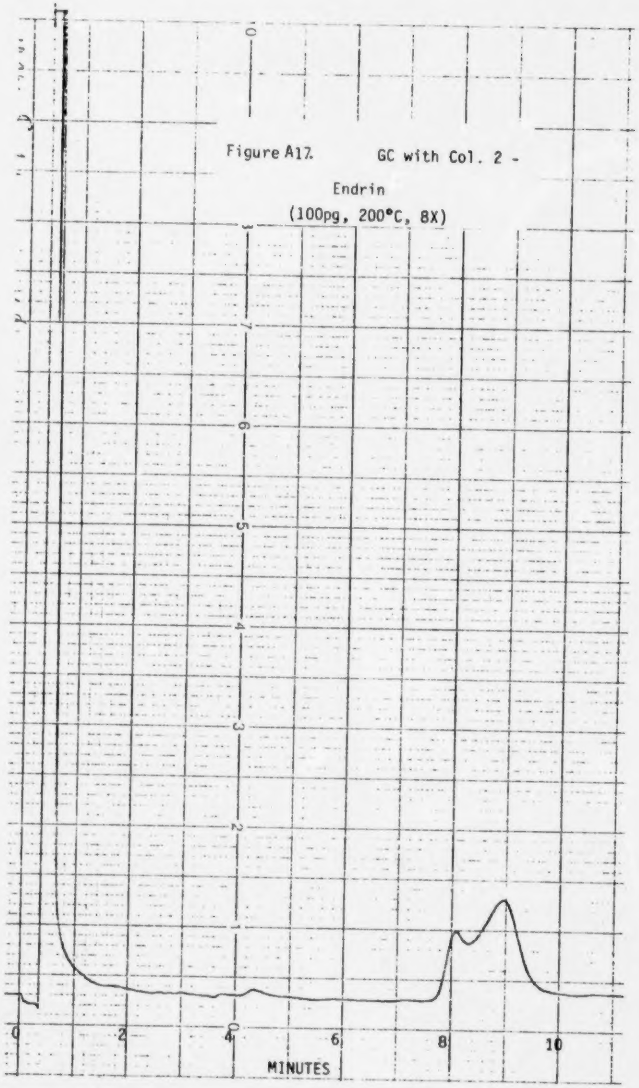


Figure A17. GC with Col. 2 - Endrin (100pg, 200°C, 8X)

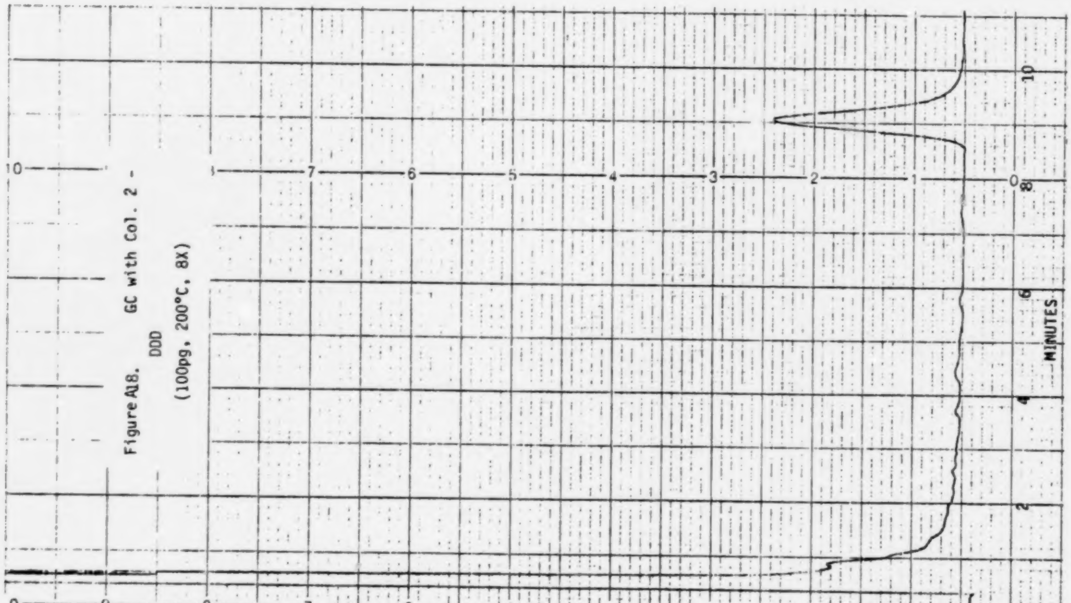


Figure A18. GC with Col. 2 - DDD (100pg, 200°C, 8X)

12-1-77 [illegible] DDD [illegible]

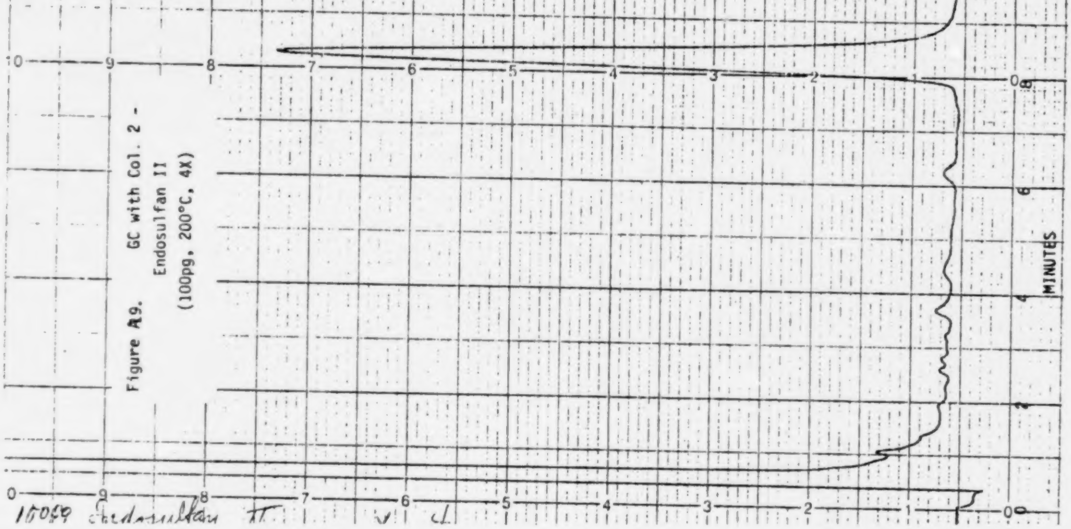


Figure A19. GC with Col. 2 - Endosulfan II (100pg, 200°C, 4X)

100pg Endosulfan II [illegible]

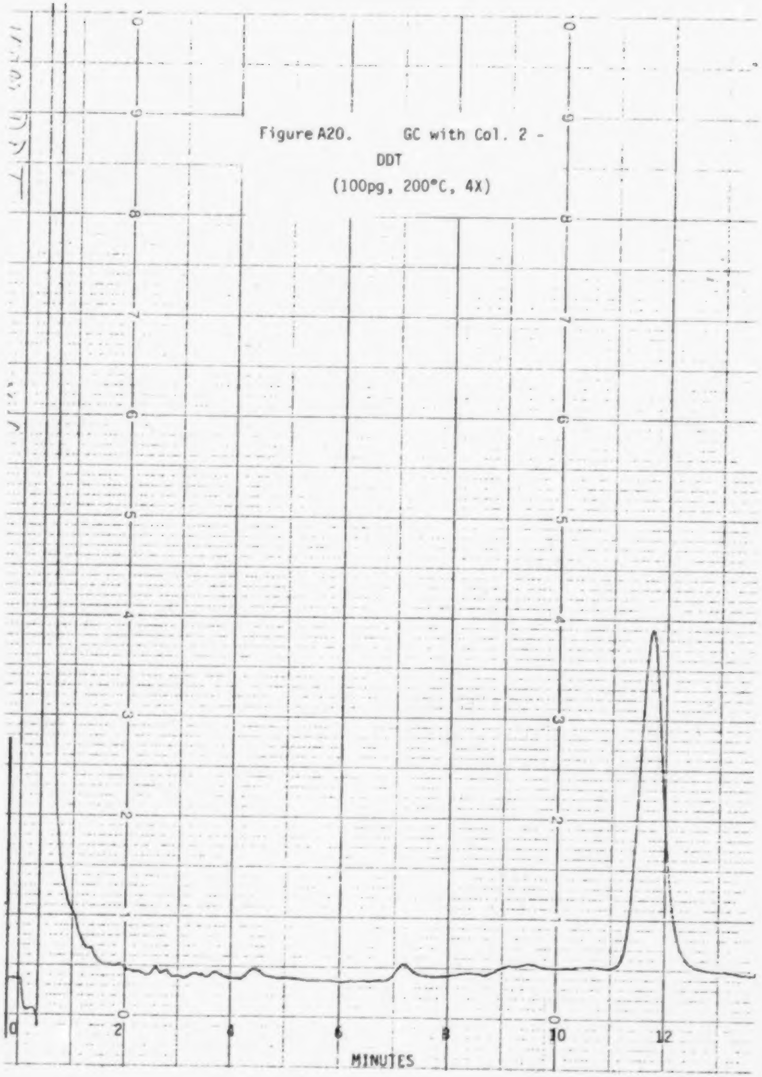


Figure A20. GC with Col. 2 - DDT (100pg, 200°C, 4X)

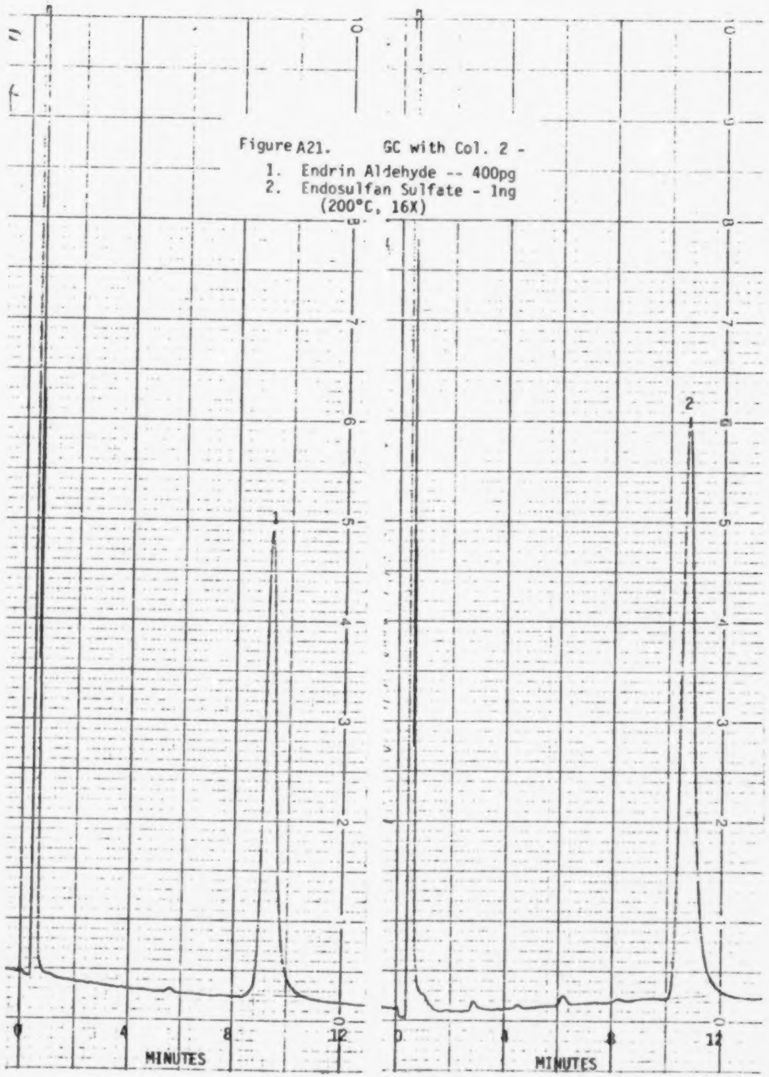


Figure A21. GC with Col. 2 -

1. Endrin Aldehyde -- 400pg
2. Endosulfan Sulfate - 1ng

(200°C, 16X)

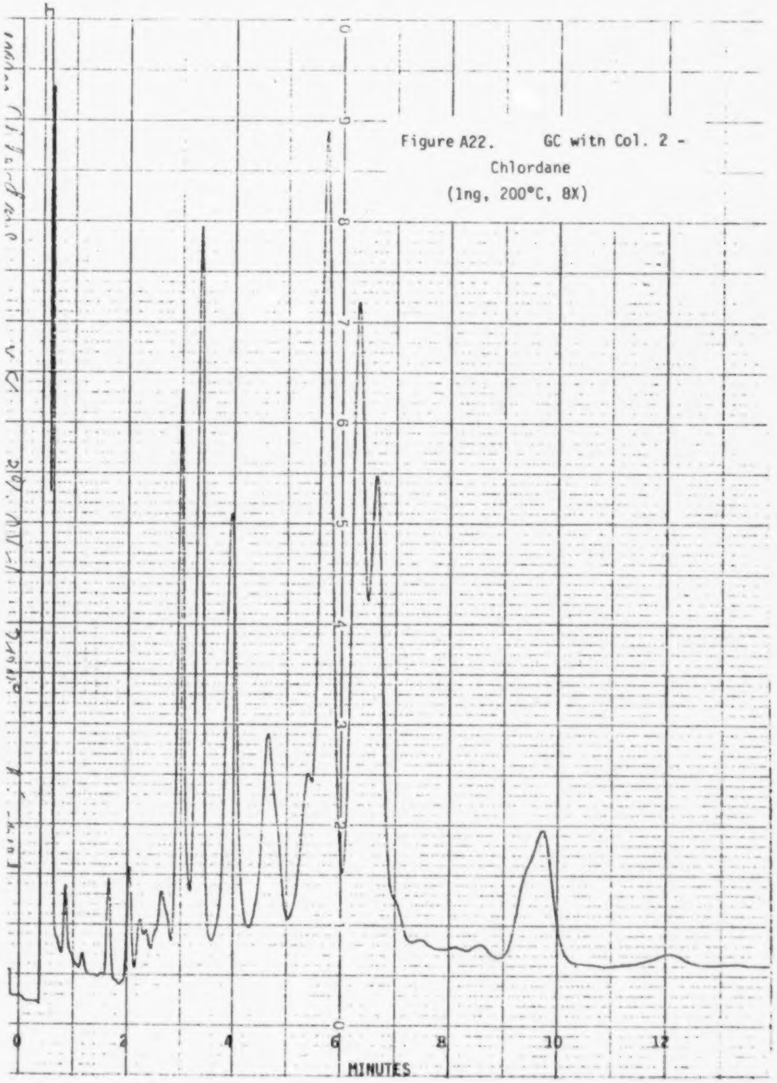


Figure A22. GC witn Col. 2 - Chlordane (1ng, 200°C, 8X)

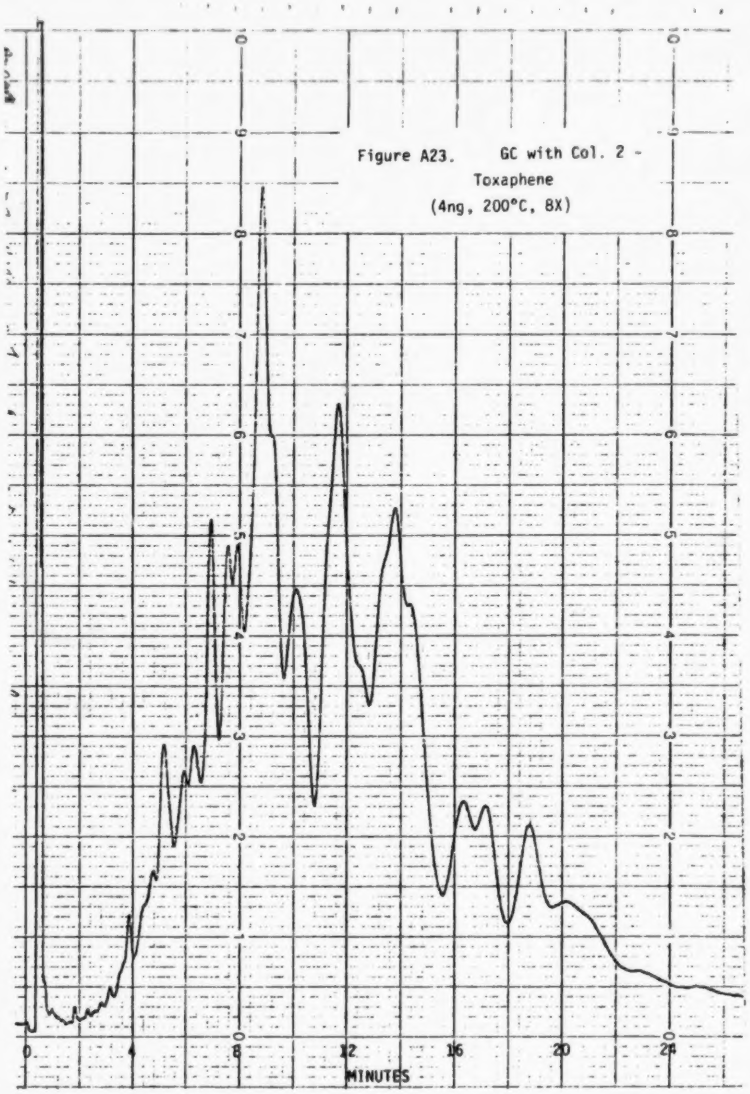


Figure A23. GC with Col. 2 -
Toxaphene
(4ng, 200°C, 8X)

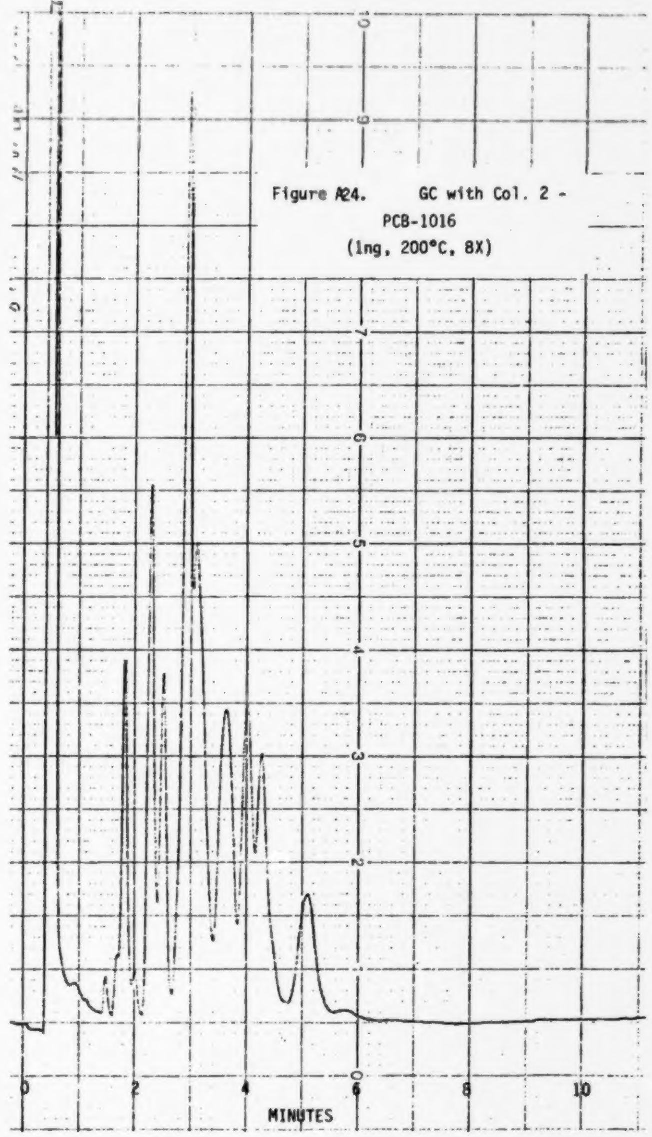


Figure A24. GC with Col. 2 -
PCB-1016
(1ng, 200°C, 8X)

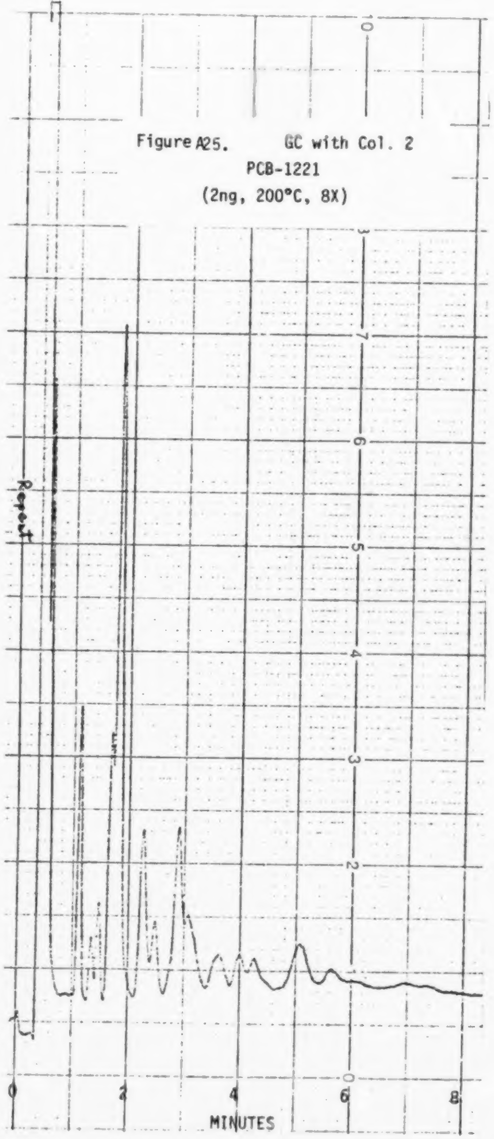


Figure A25. GC with Col. 2
PCB-1221
(2ng, 200°C, 8X)

Repeat

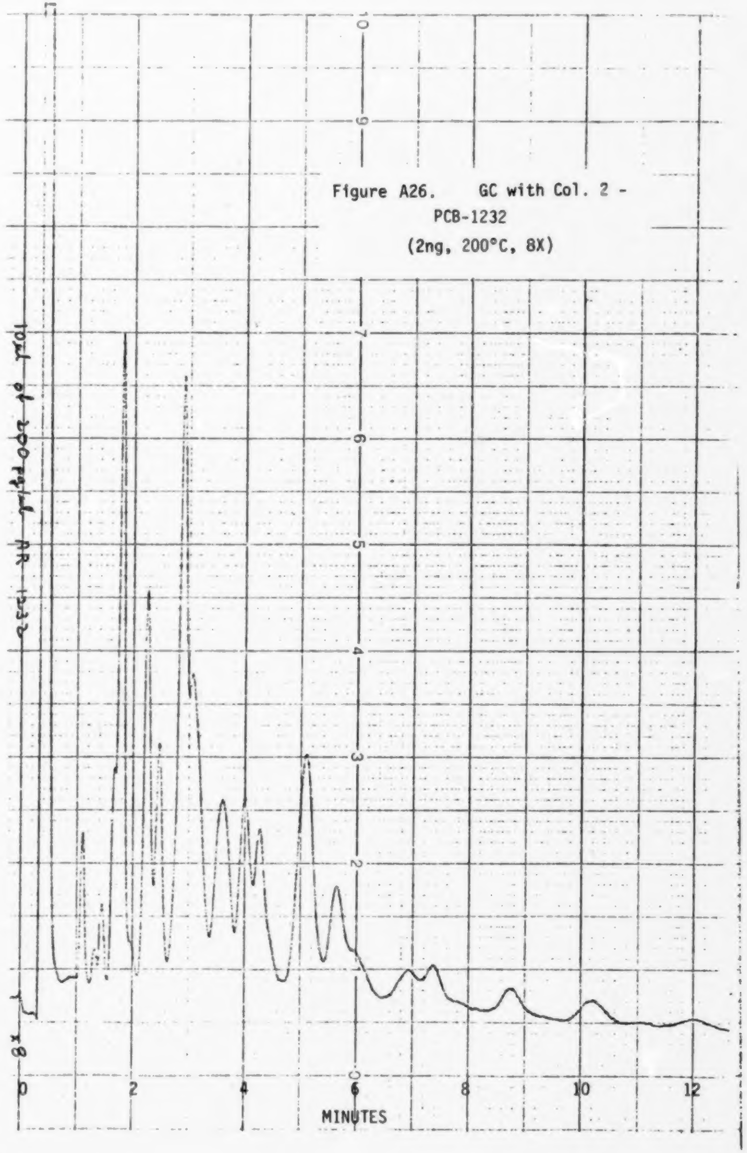


Figure A26. GC with Col. 2 -
PCB-1232
(2ng, 200°C, 8X)

10µl of 200 pg/µl AR 1232

x8

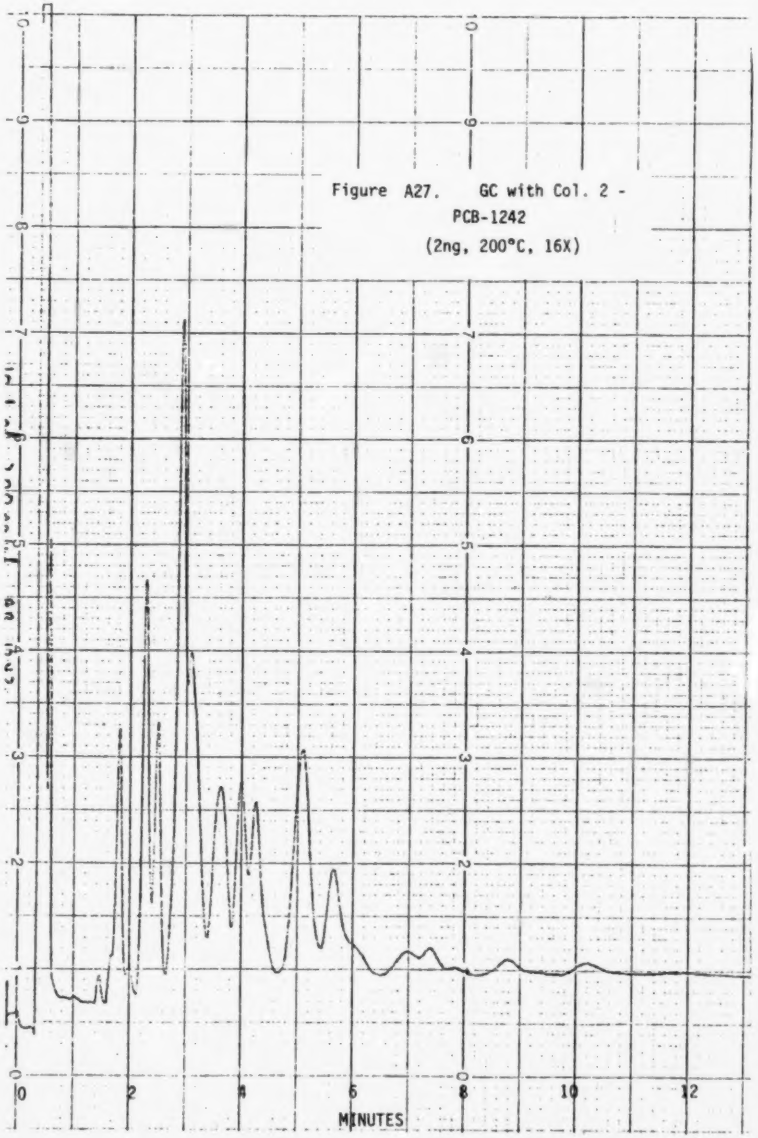


Figure A27. GC with Col. 2 -
PCB-1242
(2ng, 200°C, 16X)

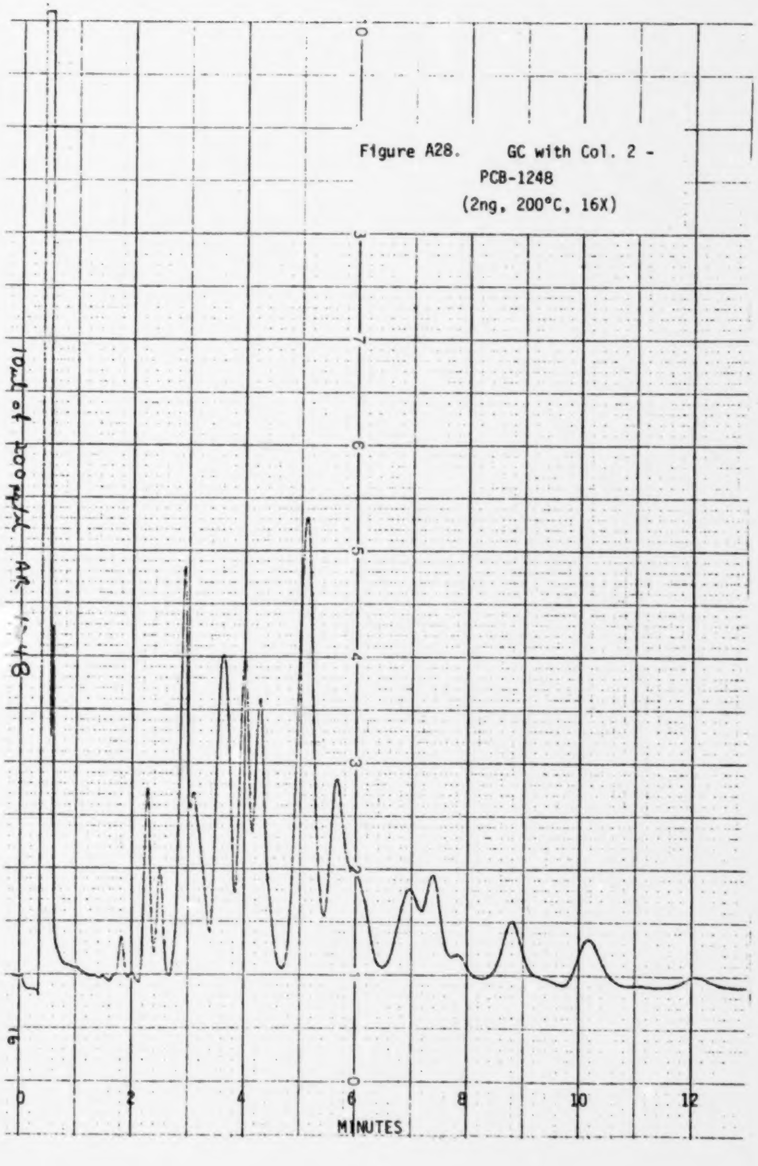


Figure A28. GC with Col. 2 -
PCB-1248
(2ng, 200°C, 16X)

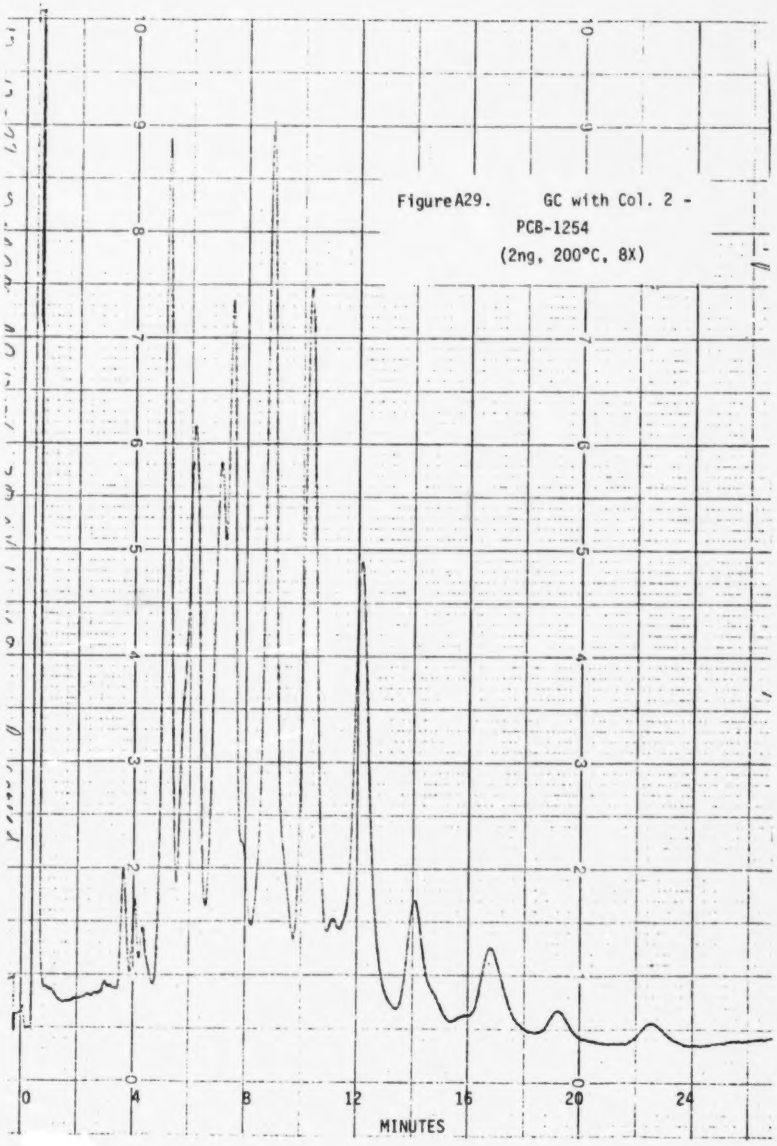


Figure A29. GC with Col. 2 - PCB-1254 (2ng, 200°C, 8X)

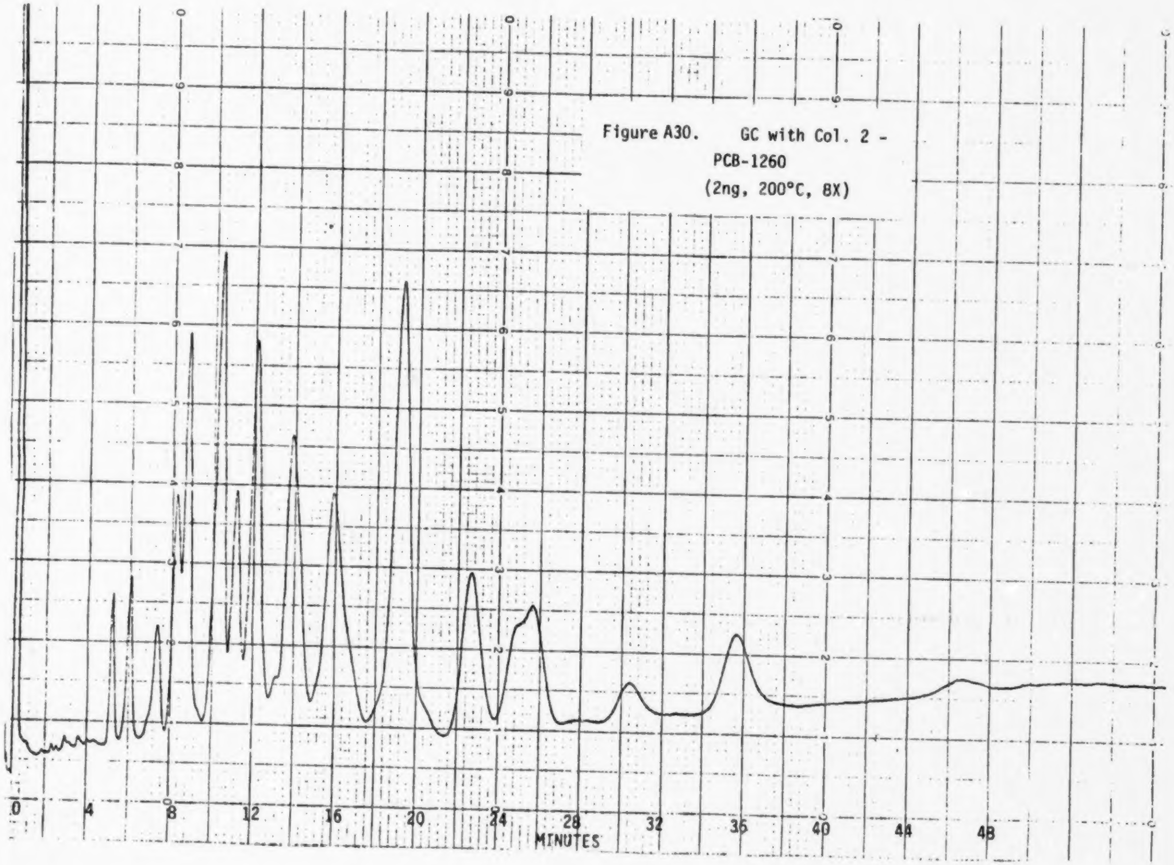


Figure A30. GC with Col. 2 -
PCB-1260
(2ng, 200°C, 8X)

APPENDIX B

EXTRACTION STUDY FIGURES AND TABLES

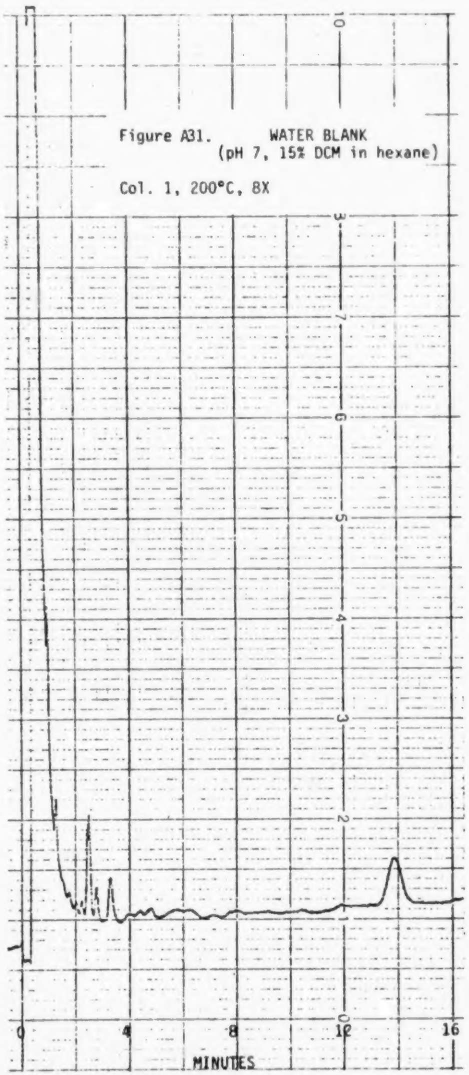


Figure A31. WATER BLANK
(pH 7, 15% DCM in hexane)

Col. 1, 200°C, 8X

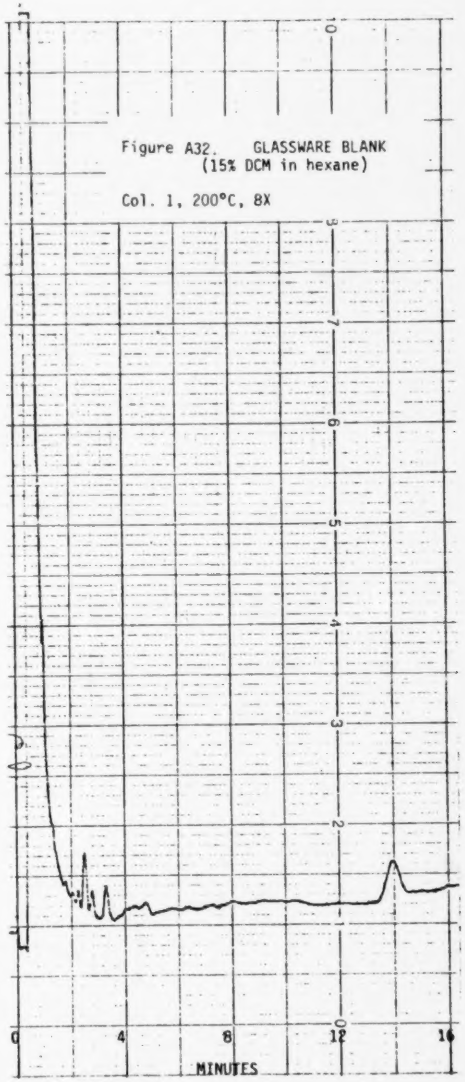


Figure A32. GLASSWARE BLANK
(15% DCM in hexane)

Col. 1, 200°C, 8X

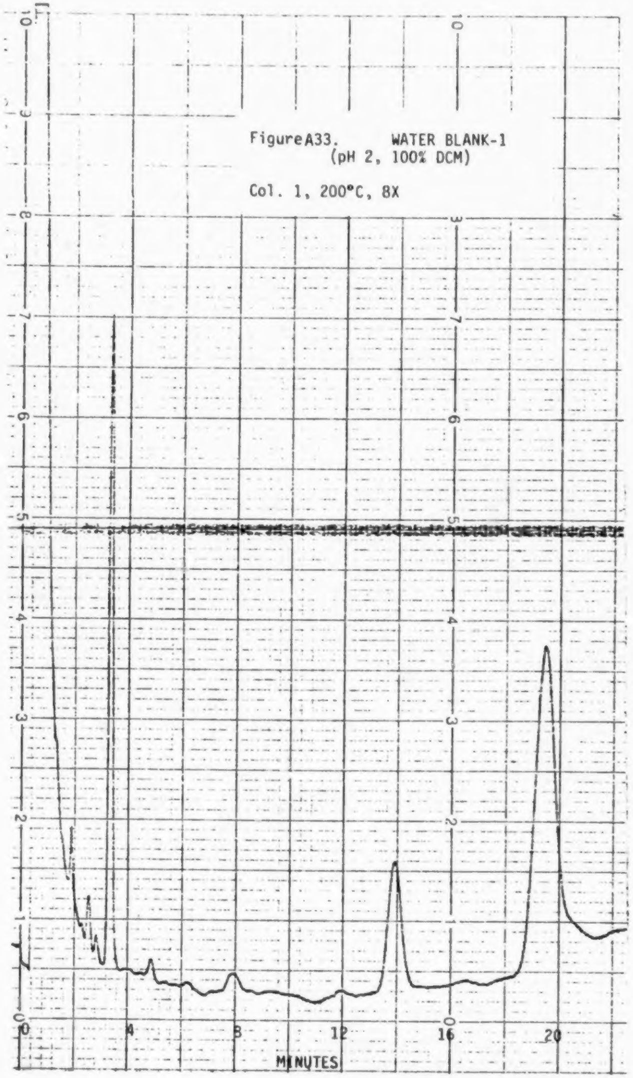


Figure A33. WATER BLANK-1
(pH 2, 100% DCM)

Col. 1, 200°C, 8X

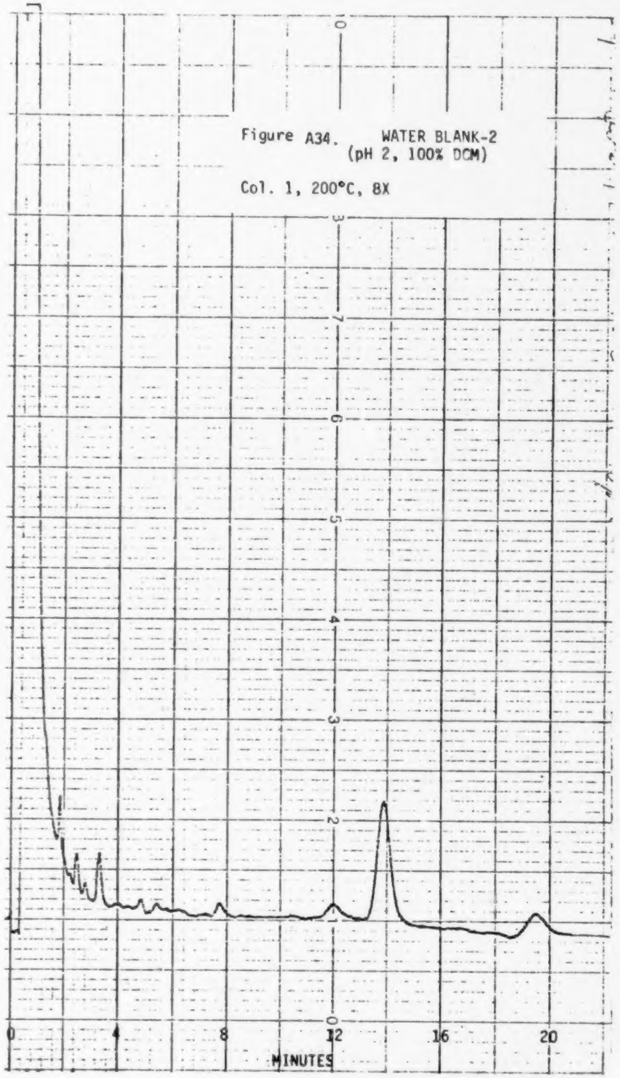


Figure A34. WATER BLANK-2
(pH 2, 100% DCM)

Col. 1, 200°C, 8X

Table A1.

EXTRACTION STUDY RESULTS - GROUP I
(percent recovery)

Solvent	pH	Replicate	a-BHC	b-BHC	d-BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
DCM	2	1	95	94	98	97	91	94	98	90
		2	98	94	106	100	98	99	104	102
		3	97	95	108	102	94	98	104	101
	7	1	92	92	91	102	93	96	103	92
		2	94	99	99	99	93	100	106	92
		3	94	96	94	97	88	95	104	96
		4	98	94	85	102	95	98	104	88
	10	1	90	92	110	84	66	89	85	87
		2	86	85	79	87	86	91	93	84
		3	100	97	101	95	91	99	99	96
DCM-Hexane	2	1	95	101	91	94	93	100	103	88
		2	95	99	88	95	94	97	103	93
		3	94	102	105	90	92	97	107	101
	7	1	93	101	87	93	93	101	99	84
		2	103	107	100	103	101	110	114	105
		3	99	104	95	100	97	104	102	88
		4	97	102	91	98	94	96	103	89
	10	1	95	102	99	93	78	99	99	83
		2	99	105	107	109	88	105	108	104
		3	99	104	103	95	90	104	107	100

	Blank Values (Subtracted)			
	delta BHC		Endosulfan Sulfate	
	15% DCM	100% DCM	15% DCM	100% DCM
pH 2	32	0	20	36
pH 7	34, 29	15	25, 15	33
pH 10	31	37	34	45

Table A2.

EXTRACTION STUDY RESULTS - GROUP II
(percent recovery)

Solvent	pH	Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
DCM	2	1	101	69	75	98	99	102	100	99
		2	114	49	53	96	93	108	98	99
		3	113	60	63	100	97	108	97	103
	7	1	87	66	56	100	100	107	99	91
		2	95	46	34	98	98	105	97	91
		3	89	66	51	103	100	106	99	88
		4	96	63	43	105	103	114	101	97
	10	1	102	75	71	105	103	109	97	107
		2	98	64	59	99	99	107	94	98
		3	102	76	74	98	101	108	92	92
DCM-Hexane	2	1	109	79	73	102	101	111	101	102
		2	103	60	50	99	97	99	98	98
		3	105	70	57	106	99	103	102	105
	7	1	111	68	57	106	105	109	107	89
		2	103	46	42	93	92	97	101	92
		3	105	78	72	108	104	108	103	92
		4	105	59	45	109	100	103	102	91
	10	1	90	79	73	101	105	105	102	111
		2	84	71	69	102	100	102	98	107
		3	85	80	78	109	102	101	96	98

	Blank Values (Subtracted)	
	15% DCM	100% DCM
pH 2 gamma BHC	0	17%
pH 7 gamma BHC	0	23%
pH 10 gamma BHC	18%	14%
Heptachlor	3%	3%

Table A3.

EXTRACTION STUDY RESULTS - CHLORDANE
(percent recovery)

Solvent	pH	Replicate	Peaks 1,2,3 i	Peak 4 (gamma)	Peak 5 (alpha)	Peak 6
DCM	2	1		101	110	101
		2		98	104	97
		3		103	110	101
	7	1		97	104	98p
		2		93	99	90p
		3		102	108	98p
		4		100	105	96p
	10	1		104	101c	98
		2		105	100c	97
		3		106	102c	105
DCM-Hexane	2	1		102	101	100p
		2		103	105	106p
		3		102	104	106p
	7	1		103	107	103p
		2		99	103	100p
		3		100	103	100p
		4		97	98	100p
	10	1		102	103	100p
		2		103	102	103p
		3		104	103	109p

i - Prohibitive interference

c - Blank value amounting to 6% subtracted from printout value.

p - Value obtained by peak height measurement.

Table A4.

EXTRACTION STUDY RESULTS - TOXAPHENE
(percent recovery)

Solvent	pH	Replicate	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6
DCM	2	1	95	94c	93	95	91	96
		2	95	91c	99	98	94	97
		3	93	89c	91	91	87	90
	7	1	99	107	100	102	99	103
		2	99	109	99	100	99	101
		3	105	103	101	120	108	108
		4	105	114	101	105	98	103
	10	1	110	128	104	110	114	110
		2	110	117	110	111	115	111
		3	109	112	105	109	110	109
DCM-Hexane	2	1	98	99	95	97	97	98
		2	100	104	100	101	100	99
		3	101	105	97	100	102	100
	7	1	99	94	98	98	99	99
		2	92	97	95	92	91	92
		3	101	103	98	99	100	100
		4	110	114	109	109	110	111
	10	1	105	95	100	106	104	105
		2	106	97	93	106	103	105
		3	105	97	93	105	102	104

c - Blank amounting to 10% subtracted from printout value.

Table A5.

EXTRACTION STUDY RESULTS - PCB-1254
(percent recovery)

Solvent	pH	Replicate	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7
DCM	2	1	77	84	83	82	87	87	79
		2	89	98	97	94	101	98	100
		3	88	95	95	91	97	96	92
	7	1	105	104	104	104	108	106	106
		2	98	98	98	96	100	100	98
		3	99	100	103	101	107	103	101
		4	94	94	99	98	98	100	99
	10	1	97	92c	95	93	103	96	101
		2	92	90c	96	93	96	96	97
		3	96	92c	99	96	99	99	99
DCM-Hexane	2	1	102	100	101	100	110	100	99
		2	108	101	104	104	113	104	106
		3	94	110	102	100	110	99	104
	7	1	96	104	100	99	102	100	100
		2	97	97	103	100	100	100	100
		3	100	101	103	103	105	103	103
		4	99	102	104	101	107	103	105
	10	1	97	97	99	98	112	97	100
		2	104	102	106	105	107	104	104
		3	109	100	104	99	109	103	102

c - Blank amounting to 6% subtracted from printout value.

Table A6.

EXTRACTION STUDY RESULTS - PCB-1016
(percent recovery)

Solvent	pH	Replicate	Peak 1_p	Peak 2_p	Peak 3_p	Peak 4_p	Peak 5_i	Peak 6_p	Peak 7_p	Peak 8_p
DCM	2	1	i	87	88	93		101	90	93
		2	i	81	90	90		96	84	86
		3	i	81	90	90		91	83	83
	7	1	92	88	91	91		98	90	85
		2	86	83	87	88		93	86	86
		3	76	77	82	83		85	79	76
		4	87	86	90	90		96	90	88
	10	1	i	93	97	96		95	97	96
		2	94	82	89	90		90	84	77
		3	89	78	82	82		88	77	77
DCM-Hexane	2	1*								
		2	i	78	82	84		86	80	79
		3	i	86	91	93		93	84	85
	7	1	97	92	92	90		98	94	101
		2	91	91	91	90		97	94	100
		3	94	95	96	95		100	96	102
		4	87	89	89	90		94	86	84
	10	1	91	77	78	81		86	79	81
		2	95	82	83	84		88	83	83
		3	90	72	73	75		80	73	78

p - peak height measurement

i - prohibitive interference

* - sample spilled after initial analysis

Table A7.

EXTRACTION STUDY RESULTS - PCB-1260
(percent recovery)

Solvent	pH	Replicate	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8	Peak 9i	Peak 10	Peak 11
DCM	2	1	89	92	95	94	97	95	100	98		100p	100p
		2	97	100	102	100	102	99	103	100		100p	99p
		3	103	108	107	107	108	106	110	104		106p	105p
	7	1	i	i	97	96	96	92	94	104		95p	99p
		2	i	i	102	101	102	96	99	i		100p	99p
		3	104	108	105	104	104	104	105	103		103p	103p
		4	108	100	98	98	100	99	100	98		99p	100p
	10	1	106	91c	109	108	109	106	107	105		105p	103p
		2	100	91c	106	106	107	104	106	104		104p	100p
		3	91	77c	93	95	96	95	96	95		97p	92p
DCM-Hexane	2	1	89	90	93	96	99	95	99	98		98p	101p
		2	89	104	104	106	109	106	110	109		109p	107p
		3	87	101	101	102	105	103	107	106		105p	104p
	7	1	97	102	100	101	102	100	102	100		100p	101p
		2	90	100	93	96	99	97	97	93		97p	98p
		3	100	104	101	101	101	102	102	100		101p	97p
		4	98	103	98	99	100	100	100	101		100p	96p
	10	1	95	101	100	99	99	98	100	98		97p	99p
		2	101	99	100	99	99	98	100	98		96p	93p
		3	96	101	96	98	101	99	99	97		99p	101p

i - Prohibitive interference.

c - Blank amounting to 17% subtracted form printout value.

p - Value obtained by peak height measurement.

Table A8.

EXTRACTION STUDY RESULTS - PCB-1221
(percent recovery)

Solvent	pH	Replicate	Peak 1_p	Peak 2_i	Peak 3_p	Peak 4_p	Peak 5_p	Peak 6_p	Peak 7_p
DCM	2	1	91		93	88	90	91	96
		2	70		73	69	72	72	76
		3	65		73	65	69	67	68
	7	1	101		100	103	102	95	109
		2	95		98	91	93	90	97
		3	94		100	100	97	100	108
		4	83		92	90	89	93	93
	10	1	81		87	81	84	89	86
		2	77		75	70	74	73	74
		3	83		79	76	80	82	83
DCM-Hexane	2	1	86		90	86	85	87	85
		2	68		71	67	66	65	67
		3	74		73	70	71	71	68
	7	1	85		83	84	85	91	87
		2	88		93	92	91	101	94
		3	86		83	84	88	88	83
		4	84		83	84	85	84	83
	10	1	58		67	56	56	54	56
		2	72		76	68	69	71	67
		3	70		68	63	65	62	67

p - value obtained by peak height measurement.

i - prohibitive interference.

Table A9.

EXTRACTION STUDY RESULTS - PCB-1232
(percent recovery)

Solvent	pH	Replicate	Peak 1_p	Peak 2_p	Peak 3_p	Peak 4_p	Peak 5_p	Peak 6_p	Peak 7_p	Peak 8_p	Peak 9_p	Peak 10_p
DCM	2	1	90	93	95	89	94	92	109	102	96	102
		2	106	93	98	94	98	94	105	104	92	111
		3	76	73	75	71	74	75	94	81	83	96
	7	1	73	75	75	73	77	77	82	80	84	93
		2	84	88	87	88	87	88	89	89	89	86
		3	78	80	81	77	80	81	86	82	84	91
		4	85	88	88	87	88	85	92	91	84	*
	10	1	91	91	93	89	93	88	104	97	90	100
		2	80	82	84	81	85	86	93	85	84	91
		3	68	65	66	62	64	66	78	67	68	85
DCM-Hexane	2	1	66	59	63	61	64	64	84	71	81	90
		2	92	81	84	82	85	83	106	89	87	99
		3	82	81	85	82	85	83	95	89	91	98
	7	1	92	100	98	100	100	100	105	110	101	100
		2	84	92	91	94	96	94	101	102	97	96
		3	81	88	81	89	91	92	97	90	106	93
		4	85	93	92	93	91	97	102	101	*	*
	10	1	81	84	87	87	89	87	102	87	91	98
		2	76	85	87	90	93	89	102	93	91	99
		3	82	84	85	84	86	85	94	85	86	95

p - peak height measurement

* - a value was not obtained due to program time error

Table A10.

EXTRACTION STUDY RESULTS - PCB-1242
(percent recovery)

Solvent	pH	Replicate	Peak 1_p	Peak 2_p	Peak 3_p	Peak 4_p	Peak 5_p	Peak 6_p	Peak 7_p	Peak 8_p	Peak 9_p
DCM	2	1	103	103	104	105	105	107	102	98	98
		2	94	96	97	98	97	101	96	86	97
		3	100	101	101	100	101	103	100	95	97
	7	1	107	99	100	97	101	104	98	98	104
		2	104	97	99	96	101	103	96	89	98
		3	106	102	103	97	107	106	104	97	98
		4	105	104	103	103	109	106	103	100	104
	10	1	106	103	103	105	106	109	103	108	98
		2	102	103	102	103	106	105	104	107	105
		3	105	103	104	103	102	107	104	104	98
DCM-Hexane	2	1	99	101	101	101	104	104	100	100	105
		2	98	101	102	100	103	104	101	98	103
		3	99	102	104	102	103	107	103	103	106
	7	1	100	99	101	99	105	102	98	97	94
		2	95	95	100	96	i	100	102	99	i
		3	103	97	99	94	105	93	97	97	106
		4	101	103	105	104	i	106	101	105	103
	10	1	99	97	99	94	104	103	96	97	98
		2	93	93	93	87	i	101	92	89	88
		3	99	97	98	95	99	104	98	97	97

p - peak height measurement

i - prohibitive interference

Table A11.

EXTRACTION STUDY RESULTS - PCB-1248
(percent recovery)

Solvent	pH	Replicate	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7
DCM	2	1	96	98	98	103	96	92	98
		2	92	96	93	98	90	96	95
		3	93	97	98	100	92	96	93
	7	1	85	93	85	95	94	97	95
		2	85	93	85	95	93	97	95
		3	90	93	88	93	92	92	95
		4	92	93	91	96	94	96	96
	10	1	92	95	89	95	95	98	97p
		2	89	86	79	86	84	89	86p
		3	90	96	91	95	94	94	96p
DCM-Hexane	2	1	92	97	97	96	95	91	95p
		2*							
		3	94	98	98	97	98	91	104p
	7	1	99	103	101	101	103	100	100
		2	95	98	88	95	98	90	97
		3	98	103	90	100	103	98	100
		4	94	96	90	94	96	93	98
	10	1	98	97	96	100	100	97	98
		2	93	95	91	96	96	94	97
		3	94	89	87	90	90	89	90p

p - peak height measurement

* Sample was accidentally contaminated after initial analysis

Table A12
EXTRACTION STUDY SUMMARY

Material	Solvent	pH	Remarks
Single-compound pesticides	15% DCM	basic	
Chlordane	either	existing	
Toxaphene	15% DCM	existing	Slight preference for acidic
PCB-1254	either	existing	Slight preference for basic
PCB-1016	15% DCM	neutral	
PCB-1260	15% DCM	basic	
PCB-1221	DCM	neutral	
PCB-1232	15% DCM	neutral-basic	Not as significant, subjective evaluation
PCB-1242	either	existing	
PCB-1248	either	existing	Slight preference for 15% DCM

Table A13
MEAN PERCENT RECOVERIES
(15% DCM, pH 10)

Compound	Recovery
α-BHC	96
β-BHC	98
δ-BHC	97
Heptachlor epoxide	99
DDE	83
DDD	101
DDT	104
Endosulfan sulfate	93
γ-BHC	86
Heptachlor	74
Aldrin	71
Endosulfan I	104
Dieldrin	100
Endrin	106
Endosulfan II	99
Endrin Aldehyde	102

Table A14
MEAN DEVIATIONS
(15% DCM, pH 10)

Material	Deviation	Average Recovery
Chlordane	±3	97-103
Toxaphene	±4	96-104
PCB-1254	±4	96-104
PCB-1016	-17	83
PCB-1260	±2	98-102
PCB-1221	-24	76
PCB-1232	-13	87
PCB-1242	±3	97 - 103
PCB-1248	-5	95

APPENDIX C

PRESERVATION STUDY FIGURES AND TABLES

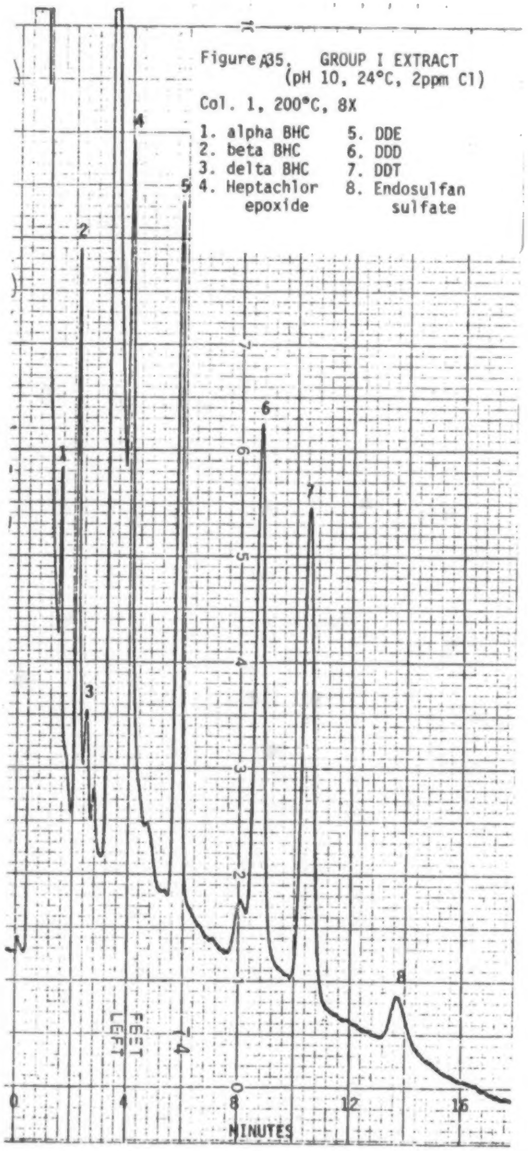


Figure A35. GROUP I EXTRACT (pH 10, 24°C, 2ppm Cl)

Col. 1, 200°C, 8X

1. alpha BHC
2. beta BHC
3. delta BHC
4. Heptachlor epoxide
5. DDE
6. DDD
7. DDT
8. Endosulfan sulfate

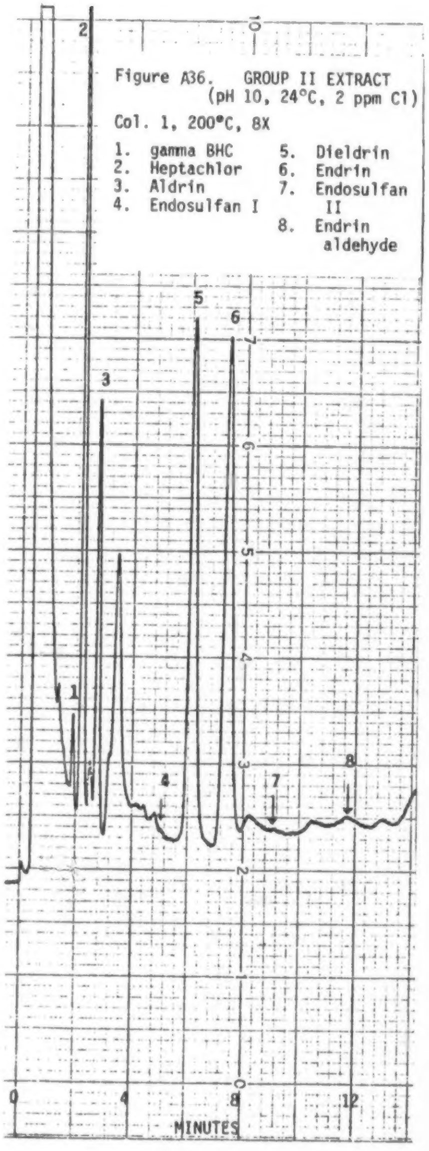


Figure A36. GROUP II EXTRACT (pH 10, 24°C, 2 ppm Cl)

Col. 1, 200°C, 8X

1. gamma BHC
2. Heptachlor
3. Aldrin
4. Endosulfan I
5. Dieldrin
6. Endrin
7. Endosulfan II
8. Endrin aldehyde

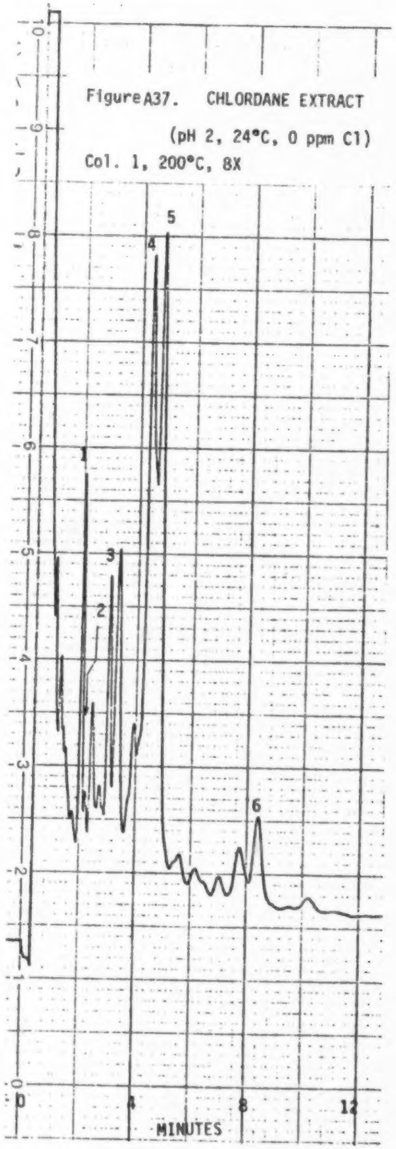


Figure A37. CHLORDANE EXTRACT (pH 2, 24°C, 0 ppm Cl)

Col. 1, 200°C, 8X

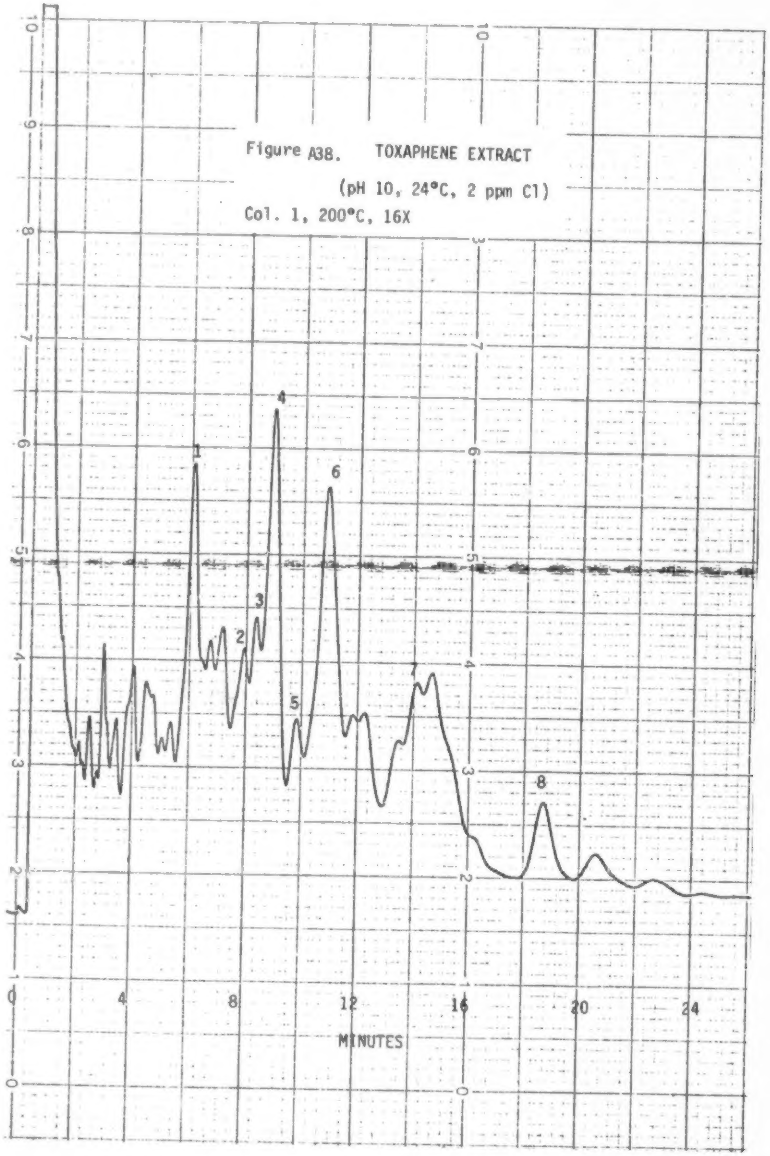


Figure A38. TOXAPHENE EXTRACT
(pH 10, 24°C, 2 ppm Cl)
Col. 1, 200°C, 16X

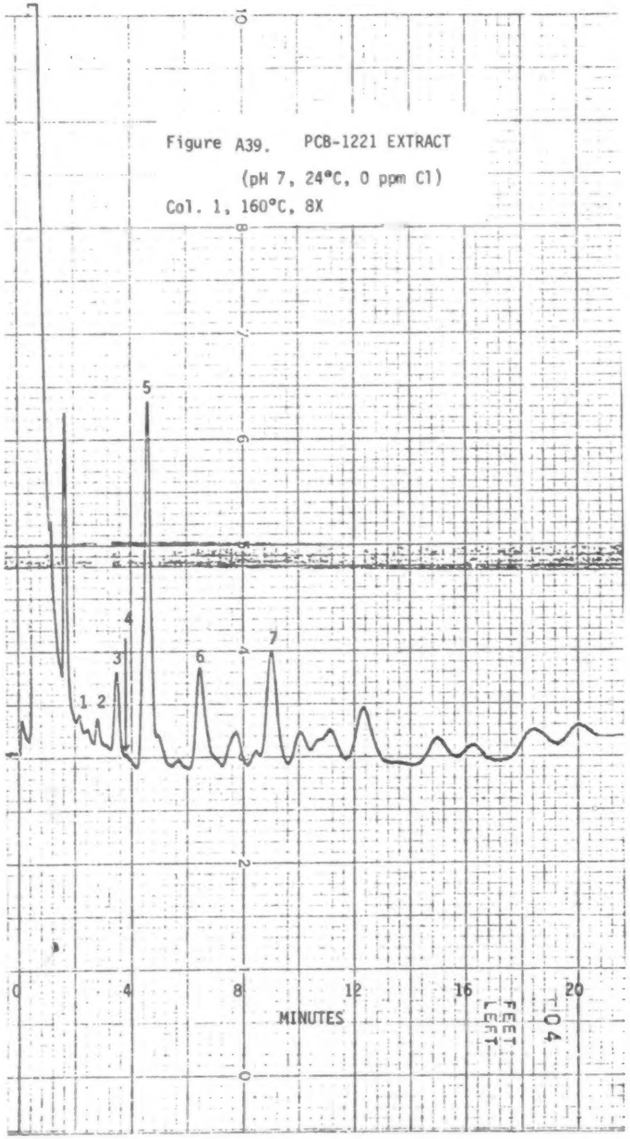


Figure A39. PCB-1221 EXTRACT

(pH 7, 24°C, 0 ppm Cl)

Col. 1, 160°C, 8X

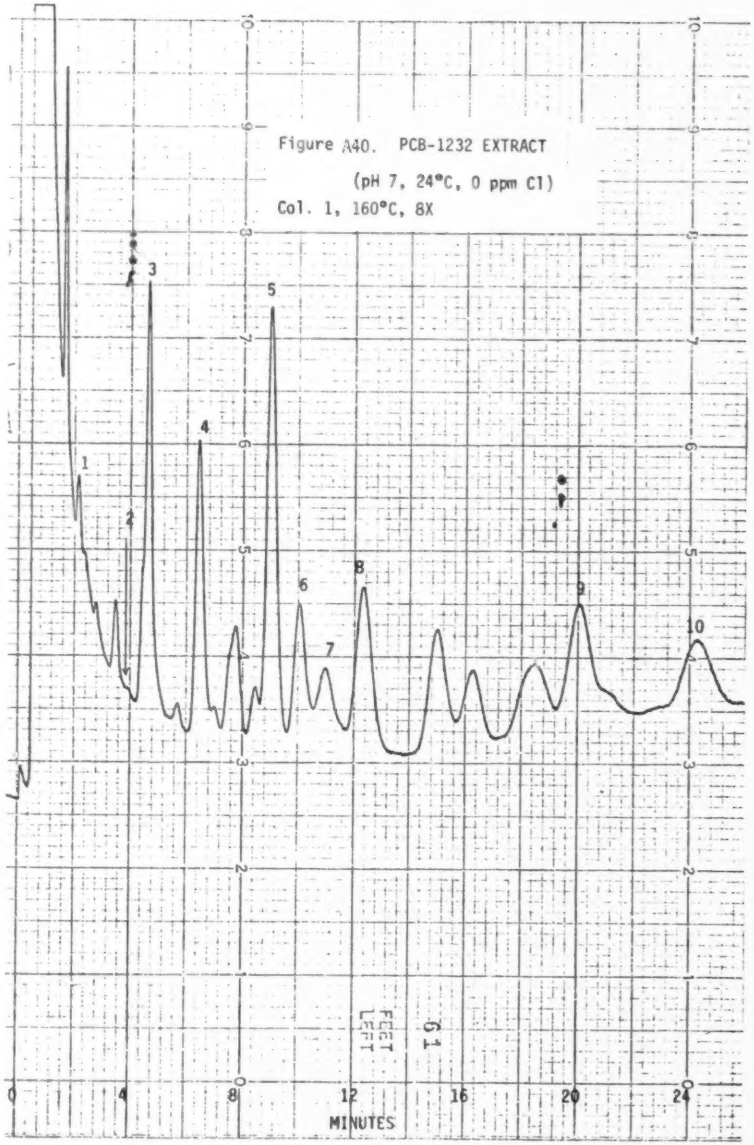


Figure A40. PCB-1232 EXTRACT

(pH 7, 24°C, 0 ppm Cl)

Col. 1, 160°C, 8X

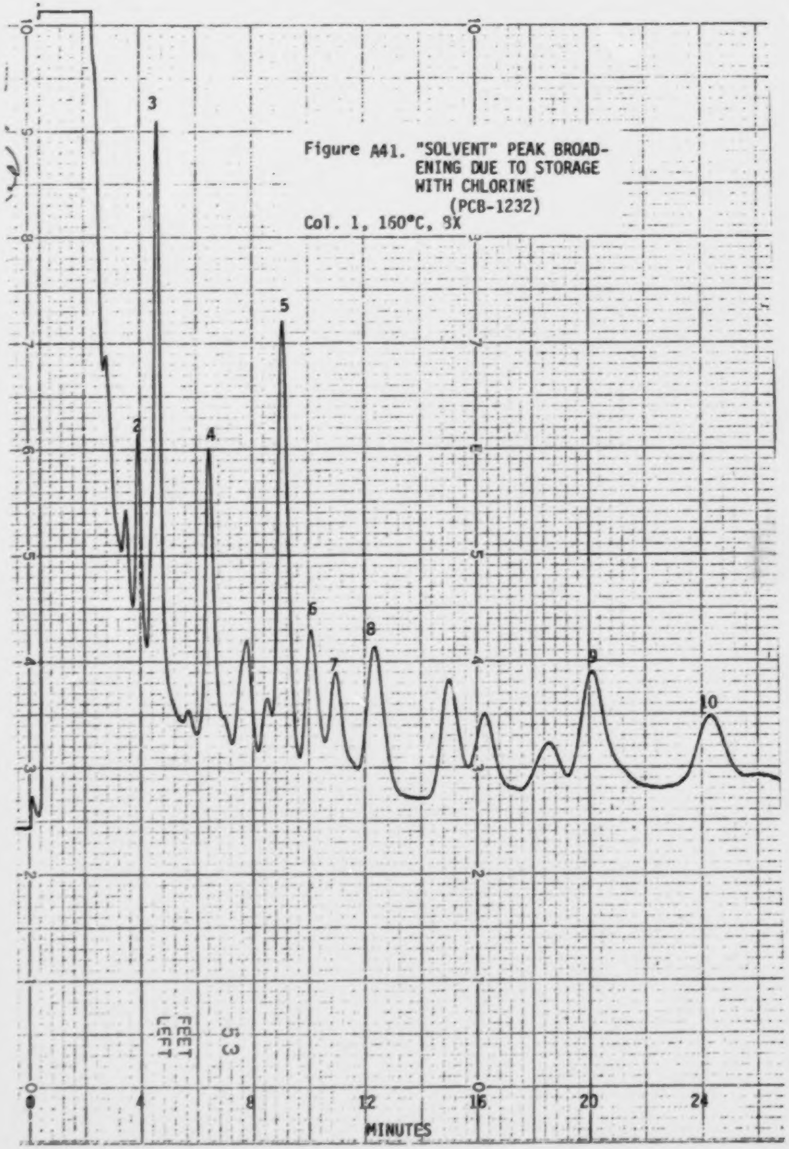


Figure A41. "SOLVENT" PEAK BROADENING DUE TO STORAGE WITH CHLORINE (PCB-1232)

Col. 1, 160°C, 3X

Table A15.

PRESERVATION STUDY RESULTS - GROUP I
(percent recovery - 7-day Storage Period)

pH	°C Temperature	ppm Chlorine	Replicate	alpha BHC	beta BHC	delta BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
2	4	0	1	71	91	97	100	99	100	98	All or most disappeared during storage period.
			2	51	76	86	93	93	92	92	
		2	1	67	97	69	97	93	96	93	
			2	67	89	63	94	90	94	91	
	24	0	1	87	102	117	177	120	115	117	
			2	69	82	32	104	103	109	102	
		2	1	50	76	64	90	81	88	84	
			2	53	76	64	95	84	91	90	
7	4	0	1	56	94	100	203	78	90	80	
			2	62	98	105	184	89	97	89	
		2	1	66	106	114	166	95	107	96	
			2	57	92	113	127	93	104	94	
	24	0	1	68	94	109	100	93	101	93	
			2	67	89	92	96	87	93	91	
		2	1	31	84	85	98	90	99	96	
			2	19	79	68	92	85	89	90	
10	4	0	1	93	66	69	114	95	95	94	
			2	80	83	74	132	94	97	96	
		2	1	79	83	74	111	87	91	89	
			2	74	90	80	116	90	97	93	
	24	0	1	27	99	36	269	120	106	85	
			2	20	84	35	200	112	94	80	
		2	1	17	95	27	188	118	96	80	
			2	10	102	25	200	119	99	80	

Table A16.

PRESERVATION STUDY RESULTS - GROUP II
(percent recovery - 7-day Storage Period)

pH	°C Temp.	ppm Chlorine	Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
2	4	0	1	100	80	88	99	105	90	100	89
			2	98	82	90	95	104	89	110	94
		2	1	101	80	15	95	103	92	106	84
			2	102	86	15	95	102	95	103	84
	24	0	1	92	14	86	102	95	21	92	91
			2	100	15	96	106	101	22	91	96
		2	1	91	13	24	75	89	24	78	64
			2	94	13	21	81	94	26	81	77
7	4	0	1	107	97	124	117	125	100	96	94
			2	99	85	112	102	107	107	107	96
		2	1	97	93	12	97	99	100	96	83
			2	96	89	11	96	98	101	94	84
	24	0	1	101	15	98	90	107	102	84	90
			2	82	15	89	84	90	80	70	80
		2	1	96	73	15p	100	106	102	93	87
			2	91	95p	15	84	100	99	75	83
10	4	0	1	85	75	91	0	103	101	0	93
			2	85	78	92	0	101	97	0	104
		2	1	83	401	76	0	101	101	0	0
			2	82	400	83	0	101	97	0	0
	24	0	1	16	11	89	0	102	92	0	95
			2	10	9	81	0	85	83	0	77
		2	1	19	487	104	0	132	133	0	0
			2	15	379	82	0	105	105	0	0

p - peak height measurement

Table A17.

PRESERVATION STUDY RESULTS - CHLORDANE
(percent recovery - 7-day Storage Period)

pH	Temp. °C	Chlorine ppm	Replicate	Peak 1p	Peak 2p	Peak 3p	Peak 4	Peak 5	Peak 6
2	4	0	1	98	80	94	105	97	100
			2	84	67	81	91	89	90
		2	1	94	76	91	105	103	103
			2	98	78	96	107	102	103
	24	0	1	102	11	100	115	110	119
			2	97	11	93	109	102	105
		2	1	89	8	85	104	100	106
			2	91	9	87	106	103	108
7	4	0	1	100	92	95	133	125	115
			2	102	84	97	112	106	98
		2	1	99	80	93	106	97	96
			2	98	81	94	108	100	97
	24	0	1	83	10	82	111	108	102
			2	89	38	84	116p	131p	119p
		2	1	89	9	83	104	99	97
			2	90	8	82	104	99	97
10	4	0	1	94	76	92	103	102	98
			2	102	83	96	113	111	107
		2	1	100	79	96	109	106	103
			2	93	76	91	103	100	95
	24	0	1	96	11	90	121	112	107
			2	95	12	87	123	116	112
		2	1	86	9	79	108	104	95
			2	99	10	78	125	115	115

p - peak height measurement.

Table A18.

PRESERVATION STUDY RESULTS - TOXAPHENE
(percent recovery - 7-day Storage Period)

pH	°C Temperature	ppm Chlorine	Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
2	4	0	1	98	114	103	100	99	100
			2	97	115	94	98	98	101
		2	1	102	121	108	103	107	103
			2	96	112	100	98	99	98
	24	0	1	99	124	95	100	89	99
			2	98	125	97	99	89	99
		2	1	91	121	91	93	86	91
			2	104	132	102	105	95	104
7	4	0	1	101	118	107	103	103	103
			2	93	112	97	95	91	99
		2	1	98	108	101	99	96	99
			2	96	106	99	98	95	97
	24	0	1	103	130	98	104	85	99
			2	100	125	86	98	82	97
		2	1	96	121	89	99	85	95
			2	103	130	99	105	90	101
10	4	0	1	141	125	160	162	85	115
			2	138	123	157	160	83	113
		2	1	121	105	136	141	71	96
			2	117	104	135	137	70	95
	24	0	1	192	81	159	165	64	102
			2	198	96	76	140	79	109
		2	1	178	81	166	154	67	98
			2	203	87	181	177	70	111

Table A19.

PRESERVATION STUDY RESULTS - PCB-1254
(percent recovery - 7-day Storage Period)

pH	°C Temp.	ppm Chlorine	Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
2	4	0	1	94	98	98	97	97	97	96
			2	100	108	104	103	111	104	104
		2	1	99	100	101	100	100	102	98
			2	95	96	96	95	94	96	95
	24	0	1	92	103	101	98	102	98	93
			2	94	92	98	95	99	94	88
		2	1	91	92	95	92	95	92	88
			2	90	91	96	92	97	92	89
7	4	0	1	94	98	101	100	102	97	94
			2	95	98	100	100	107	98	99
		2	1	93	92	94	94	94	93	94
			2	95	94	101	100	103	97	92
	24	0	1	88	93	98	95	102	94	95
			2	91	95	100	98	102	96	104
		2	1	90	92	98	96	102	95	95
			2	92	95	102	99	106	97	99
10	4	0	1	95	97	99	97	99	95	95
			2	95	95	97	95	91	94	94
		2	1	92	96	99	96	91	91	92
			2	92	95	98	94	95	96	94
	24	0	1	89	96	101	93	98	92	92
			2	97	93	101	96	96	95	92
		2	1	91	92	94	94	94	93	93
			2	91	95	97	94	97	94	101

Table A20.

PRESERVATION STUDY RESULTS - PCB-1016
(percent recovery - 7-day Storage Period)

pH	Temp. °C	Chlorine ppm	Replicate	Peak 1	Peak 2p	Peak 3	Peak 4	Peak 5p	Peak 6	Peak 7p	Peak 8
2	4	0	1	93	93	95	93	112	95	95	96
			2	92	93	93	92	106	91	93	94
		2	1	95	99	97	96	115	94	98	91
			2	97	100	103	97	118	100	100	97
	24	0	1	95	94	94	90	116	98	97	91
			2	91	93	94	89	124	101	97	94
		2	1	88	92	94	83	117	102	90	89
			2	88	94	94	88	120	100	92	96
7	4	0	1	95	94	94	89	105	98	94	96
			2	92	93	92	91	107	98	97	91
		2	1	90	95	95	93	105	100	97	97
			2	88	93	92	93	105	96	94	89
	24	0	1	103	100	101	100p	95	99	102	100p
			2	90	97	94	94p	130	108	97	95p
		2	1	89	92	95	92p	115	101	93	92
			2	90	94	96	95p	120	102	92	90
10	4	0	1	88	93	94	88	107	104p	95	95p
			2	92	95	97	89	125	114p	100	100p
		2	1	90	93	92	91	95	97p	98	93p
			2	88	95	96	93	107	100p	97	96p
	24	0	1	91	92	97	85	107	95p	91	87p
			2	88	94	96	81	117	97p	94	89p
		2	1	85	91	96	84	93	93p	92	96p
			2	89	97	100	87	109	97p	89	83p

p - peak height measurement.

Table A21.

PRESERVATION STUDY RESULTS - PCB-1260
(percent recovery - 7-day Storage Period)

pH	Temp. °C	Chlorine ppm	Replicate	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8	Peak 9	Peak 10	Peak 11
2	4	0	1	103	158	103	101	98	91	99	84	106	89	92
			2	102	96	87	88	89	85	89	82	89	84	105
		2	1	106	106	99	99	99	98	101	93	92	98	101
			2	102	102	96	96	97	98	101	90	91	95	99
	24	0	1	101	93	98	99	98	95	82	94	84	84	88
			2	103	86	98	99	97	93	100	91	61	81	96
		2	1	89	79	94	95	93	91	96	87	75	79	87
			2	100	96	104	104	101	98	105	100	87	83	92
7	4	0	1	96	89	92	93	92	90	95	89	87	89	94
			2	93	89	89	89	90	89	91	83	83	86	84
		2	1	91	89	89	89	90	90	93	86	89	90	96
			2	97	93	92	92	92	93	96	89	89	95	97
	24	0	1	85	91	87	88	87	85	90	81	73	82	87
			2	94	102	100	99	96	96	99	88	86	90	98
		2	1	94	90	92	92	91	90	91	86	81	85	89
			2	96	91	93	91	89	89	93	85	80	84	87
10	4	0	1	94	111	88	88	87	85	88	78	96p	80	89
			2	114	132	99	98	97	92	97	82	111p	84	91
		2	1	91	95	89	87	85	85	88	76	77	77	80
			2	93	99	94	93	92	92	95	89	84	88	96
	24	0	1	87	89	91	92	90	87	90	86	68	91	89
			2	83	97	90	91	89	86	92	80	64	86	84
		2	1	89	88	92	92	90	88	92	83	72	87	81
			2	85	76	87	87	86	84	88	80	73	88	83

p - peak height measurement.

Table A22.

PRESERVATION STUDY RESULTS - PCB-1221
(percent recovery - 7-day Storage Period)

pH	Temp. °C	Chlorine ppm	Replicate	Peak 1p	Peak 2p	Peak 3p	Peak 4p	Peak 5p	Peak 6p	Peak 7p
2	4	0	1	94	96	93	93	93	92	94
			2	90	91	91	89	92	97	106
		2	1	93	145	78	117	120	150	102
			2	97	127	83	92	118	128	92
	24	0	1	89	98	91	89	89	88	88
			2	87	95	86	80	82	84	83
		2	1	94	93	83	85	98	102	86
			2	91	96	78	83	95	98	83
7	4	0	1	93	93	95	91	91	95	90
			2	91	95	93	87	90	91	92
		2	1	103	105	80	84	84	83	83
			2	113	300	89	96	97	93	98
	24	0	1	5	27	93	8	74	85	78
			2	3	32	90	0	61	84	90
		2	1	95	89	98	95	95	93	97
			2	96	103	93	92	91	91	90
10	4	0	1	93	95	91	88	92	100	123
			2	95	95	93	90	92	90	95
		2	1	91	95	93	87	90	90	94
			2	90	95	93	88	91	92	94
	24	0	1	83	93	85	82	84	83	86
			2	84	91	80	81	84	85	86
		2	1	86	93	83	81	84	83	84
			2	93	95	89	87	88	87	91

p - peak height measurement.

Table A23.

PRESERVATION STUDY RESULTS - PCB-1232
(percent recovery - 7-day Storage Period)

pH	Temp. °C	Chlorine ppm	Replicate	Peak 1p	Peak 2p	Peak 3p	Peak 4p	Peak 5p	Peak 6p	Peak 7p	Peak 8p	Peak 9p	Peak 10p
2	4	0	1	93	80	83	80	82	85	92	86	83	86
			2	97	82	85	84	87	92	113	97	90	94
		2	1	110	85	95	91	88	89	94	86	83	79
			2	112	83	92	89	88	89	94	86	86	79
	24	0	1	98	85	87	85	87	88	112	101	91	94
			2	96	83	87	87	93	100	172	135	96	118
		2	1	102	78	87	86	83	90	119	93	81	82
			2	109	78	87	84	84	87	109	90	83	88
7	4	0	1	59	80	96	94	98	95	98	97	102	86
			2	31	94	97	94	97	94	98	94	94	86
		2	1	*	91	93	92	95	92	105	97	98	92
			2	34	67	92	95	98	100	107	99	100	89
	24	0	1	23	2	64	83	83	83	66	97	83	84
			2	24	10	78	95	88	92	100	100	93	84
		2	1	93	87	90	87	87	84	100	92	88	83
			2	*	76	80	77	78	79	100	89	78	75
10	4	0	1	97	92	94	94	95	93	104	93	95	100
			2	101	92	92	93	94	96	98	101	100	97
		2	1	88	90	91	91	91	91	102	92	98	94
			2	88	91	93	92	90	96	104	94	95	106
	24	0	1	118	96	97	95	96	95	124	109	94	94
			2	116	96	96	95	97	100	129	107	98	92
		2	1	103	90	91	90	91	89	100	95	98	87
			2	97	84	86	86	87	83	89	84	85	84

* - peak not separated from "solvent" peak.

p - peak height measurement.

Table A24.

PRESERVATION STUDY RESULTS - PCB-1242
(percent recovery - 7-day Storage Period)

pH	Temp. °C	Chlorine ppm	Replicate	Peak 1p	Peak 2p	Peak 3p	Peak 4p	Peak 5p	Peak 6p	Peak 7p	Peak 8p	Peak 9p
2	4	0	1	99	97	97	99	105	95	97	100	91
			2	93	91	91	90	95	89	90	94	86
		2	1	98	95	94	97	100	90	95	96	89
			2	98	96	97	96	105	95	97	100	94
	24	0	1	93	91	92	91	107	100	97	93	97
			2	93	94	97	97	114	110	102	100	106
		2	1	93	92	92	86	102	93	93	91	94
			2	93	94	96	95	107	98	100	100	94
7	4	0	1	83	85	84	83	87	90	84	87	84
			2	84	84	84	83	87	91	86	88	89
		2	1	90	89	89	88	91	92	90	90	85
			2	91	90	90	89	91	94	93	90	88
	24	0	1	94	90	89	84	100	95	85	88	91
			2	92	88	89	84	100	95	85	86	86
		2	1	94	91	93	89	105	100	93	95	91
			2	97	93	94	100	107	103	95	95	97
10	4	0	1	101	98	98	100	108	99	98	98	103
			2	98	94	95	93	110	95	97	96	103
		2	1	99	99	97	96	100	99	95	95	100
			2	102	97	96	95	105	95	95	93	94
	24	0	1	98	95	97	95	114	113	100	100	97
			2	97	88	90	82	109	108	92	90	97
		2	1	99	94	95	91	110	101	95	93	94
			2	100	95	97	94	110	103	97	96	97

p - peak height measurement.

Table A25.

PRESERVATION STUDY RESULTS - PCB-1248
(percent recovery - 7-day Storage Period)

pH	Temp. °C	Chlorine ppm	Replicate	Peak 1p	Peak 2p	Peak 3p	Peak 4p	Peak 5p	Peak 6p	Peak 7p
2	4	0	1	90	89	90	91	91	91	91
			2	90	88	89	88	88	91	91
		2	1	98	100	98	97	97	96	95
			2	94	96	95	94	94	92	91
	24	0	1	88	86	97	93	92	90	94
			2	90	85	92	90	90	89	89
		2	1	89	89	90	90	92	90	88
			2	90	91	91	90	90	91	90
7	4	0	1	87	89	88	91	90	93	94
			2	94	98	100	95	93	96	98
		2	1	85	86	89	89	89	88	91
			2	89	89	88	93	92	92	92
	24	0	1	93	95	96	94	95	95	95
			2	93	95	94	95	94	94	91
		2	1	90	92	92	93	94	94	91
			2	89	91	91	92	91	91	93
10	4	0	1	93	94	94	96	96	95	93
			2	90	94	91	91	89	92	91
		2	1	93	94	93	94	95	93	93
			2	92	93	92	94	93	97	91
	24	0	1	87	90	93	93	92	92	94
			2	90	91	94	95	92	94	94
		2	1	85	88	91	89	89	89	90
			2	88	92	96	93	95	92	95

p - peak height measurement.

Table A26.

SUMMARY OF PRESERVATION STUDY ANALYSIS - GROUP I

	FACTOR						
Compound	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
α-BHC	2	4	0	(10,4)	(2,0)	(4,0) (4,2)	(10,4,0) (10,4,2)
β-BHC	--*	--	--	except (2,24), (10,4)	--	U+	--
δ-BHC	7	4	0	(7,4)	(2,0),(7,0) (7,2)	except (24,2)	--
DDE	2	4	0	(2,4), (2,24)	(2,2),(7,0) (7,2)	(4,0) (4,2)	U
DDD	--	--	--	--	U	U	--
DDT	2,7	--	--	except (7,4), (10,24)	(2,0)	except (24,2)	--

* Line indicates no significant difference.

+ U indicates results do not lend themselves to ready interpretation.

Mean Recoveries, %

	COMPOUND					
Conditions	α-BHC	β-BHC	δ-BHC	DDE	DDD	DDT
(2,4)	63	88	79	94	98	94
(7,4)	60	98	108	89	98	90
(10,4)	82	81	74	92	97	93

Table A27.

SUMMARY OF PRESERVATION STUDY ANALYSIS - GROUP II

	FACTOR						
Compound	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
γ-BHC	--*	4	--	--	--	--	--
Heptachlor	7	4	0	(2,4),(7,4)	(7,2)	(4,0),(4,2)	(2,4,0),(2,4,2) (7,4,0),(7,4,2)
Aldrin	--	--	0	(7,4)	(2,0),(7,0)	(4,0),(24,0)	(2,4,0),(2,24,0) (7,24,0)
Endosulfan I	--	4	0	--	--	--	(2,4,0),(2,4,2)
Dieldrin	--	--	--	--	--	--	--
Endrin	7	4	--	except (2,24)	--	--	--
Endosulfan II	--	4	--	--	--	--	--
Endrin aldehyde	--	--	0	--	--	--	(2,4,2)

* Line indicates no significant difference.

Mean Recoveries, %

	COMPOUND							
Conditions	γ-BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
pH 2, 4°C	100	82	52 (89)$^+$	96	105	92	102	84-92
pH 7, 4°C	100	91	65 (118)	103	105	102	102	84-95

$^+$ Number in parenthesis represents recovery in absence of residual chlorine.

Table A28.

SUMMARY OF PRESERVATION STUDY ANALYSIS - CHLORDANE

	FACTOR						
Peak	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
1	--*	4	--	--	--	--	--
2	--	4	--	--	--	--	--
3	--	4	--	not pH 7-10, 24°C	--	--	--
4	--	--	--	--	--	--	--
5	--	--	--	--	--	--	--
6	--	--	--	--	--	--	--

* Line indicates no significant difference.

Mean Recoveries, %
(4°C, all pH's)

Peak					
1	2	3	4	5	6
98.8	79.3	92.6	110.0	105.6	103.6

Table A29.

SUMMARY OF PRESERVATION STUDY ANALYSIS - TOXAPHENE

	FACTOR						
Peak	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
1	2,7	4	--*	(2,4),(7,4)	--	--	--
2	U⁺	--	U	U	--	--	--
3	2,7	--	--	--	--	--	--
4	2,7	--	--	--	--	--	--
5	2,7	4	--	--	--	--	--
6	--	--	--	--	--	--	--

* Line indicates no significant difference.

⁺ U indicates results do not lend themselves to ready interpretation.

Mean Recoveries, %
(pH 2,7, 4°C)

Peak

1	2	3	4	5	6
97-100	111-116	98	100	93	101

Table A30.

SUMMARY OF PRESERVATION STUDY ANALYSIS - PCB-1254

	FACTOR						
Peak	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
1	--*	4	--	--	--	--	--
2	--	4	--	--	--	--	--
3	--	--	--	--	--	--	--
4	--	4	--	--	--	--	--
5	2,7	--	--	--	--	--	--
6	--	4	--	--	--	--	--
7	--	--	--	U+	--	--	--

* Line indicates no significant difference.

+ U indicates results do not lend themselves to ready interpretation.

Mean Recoveries, %
(4°C)

Peak

1	2	3	4	5	6	7
95	97	99	98	95-102	97	94-98

Table A31.

SUMMARY OF PRESERVATION STUDY ANALYSIS - PCB-1016

	FACTOR						
Peak	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
1	--*	--	--	--	--	--	--
2	--	--	--	U+	(2,2),(7,0)	except (4,0)	--
3	--	--	--	--	--	--	--
4	7	4	--	(2,4),(7,24) (7,4)	--	(4,2)	--
5	--	--	--	--	--	--	--
6	--	--	--	U+	--	--	--
7	--	4	--	except (10,24)	--	except (24,2)	--
8	--	4	--	except (10,24)	--	--	--

* Line indicates no significant difference.

+ U indicates results do not lend themselves to ready interpretation.

Mean Recoveries, %
(pH 7, 4°C)

Peak

1	2	3	4	5	6	7	8
91.1	93.8	95.5	91.5	111.3	98.0	95.5	93.2

Table A32.

SUMMARY OF PRESERVATION STUDY ANALYSIS - PCB-1260

	FACTOR						
Peak	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
1	2	4	--*	--	--	--	--
2	--	4	--	except (2,24), (10,24)	--	--	--
3	--	--	--	--	--	--	--
4	2	--	--	--	--	--	--
5	2	--	--	--	--	--	--
6	2,7	--	--	--	--	--	--
7	--	--	--	--	--	--	--
8	2,7	--	--	--	--	--	--
9	--	4	--	--	--	(4,0),(4,2)	--
10	--	--	--	(2,4),(10,24)	--	--	--
11	2,7	4	--	--	--	--	--

* Line indicates no significant difference.

Mean Recoveries, %
(pH 2, 4°C)

Peak

1	2	3	4	5	6	7	8	9	10	11
99	102	93	98	97	94	94	90	91	92	95

Table A33.

SUMMARY OF PRESERVATION STUDY ANALYSIS - PCB-1221

	FACTOR						
Peak	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
1	2,10	4	2	except (7,4) (7,24)	U*	U	except (7,4,2), (7,24,0),(10,24,0)
2	2,10	4	0	except (2,4) (7,24)	U	U	except (2,4,0), (7,4,0),(7,4,2)
3	7,10	--[+]	0	(7,24), (10,24)	except (2,2)	(4,0)	--
4	2,10	4	2	except (7,24)	except (7,2)	except (24,0)	except (7,24,0)
5	U	4	0	except (2,4) (7,24)	except (7,0)	except (24,0)	U
6	U	U	0	except (2,4)	except (2,2)	--	except (2,4,2)
7	--	4	--	--	--	--	--

* U indicates results do not lend themselves to ready interpretation.

[+] Line indicates no significant difference.

Mean Recoveries, %
(pH 10, 4°C)

Peak

1	2	3	4	5	6	7
92	95	93	88	91	93	97

Table A34.

SUMMARY OF PRESERVATION STUDY ANALYSIS - PCB-1232

	FACTOR						
Peak	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
1	2,10	4	2	except (7,4),(7,24)	(2,0), (10,2)	U*	U
2	10	4	0	except (7,24)	except (7,0)	except (24,0)	U
3	10	4	--+	(7,4), (10,4) (10,24)	except (7,0)	--	U
4	7,10	4	--	--	--	except (24,2)	--
5	7,10	4	0	(7,4) (10,24)	--	except (24,2)	--
6	--	--	--	except (7,24)	--	except (24,2)	--
7	--	--	--	--	--	--	--
8	U	--	--	--	--	except (24,0)	--
9	7,10	--	--	(7,4),(10,4) (10,24)	--	--	--
10	--	--	--	(2,24)(10,4)	--	--	--

* U indicates results do not lend themselves to ready interpretation.

+ Line indicates no significant difference.

Mean Recoveries, %

	Peak									
Conditions	1	2	3	4	5	6	7	8	9	10
pH 7-4°C	39	83	95	91	97	93	105	94	99	88
pH 10-4°C	94	91	93	91	93	93	105	94	97	99

Table A35.

SUMMARY OF PRESERVATION STUDY ANALYSIS - PCB-1242

Peak	FACTOR						
	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
1	10	--*	2	(2,4),(10,4) (10,24)	--	--	--
2	2,10	--	2	(2,4),(10,4)	--	--	--
3	2,10	--	2	--	--	--	--
4	--	--	--	--	--	--	--
5	7	4	--	(7,4)	U+	--	--
6	2,7	4	--	--	(2,0),(7,2), (10,2)	--	--
7	2,10	--	--	--	except (7,0)	--	--
8	2,10	--	--	--	--	--	--
9	10	--	--	(2,24),(10,4) (10,24)	--	--	--

* Line indicates no significant difference.

+ U indicates results do not lend themselves to ready interpretation.

Mean Recoveries, %

Conditions	Peak								
	1	2	3	4	5	6	7	8	9
pH 2, 4°C	97	95	95	92	101	96	96	97	90
pH 7, 4°C	87	87	89	92	90	95	89	90	87
pH 10, 4°C	100	97	96	92	106	102	96	95	100

Table A36.

SUMMARY OF PRESERVATION STUDY ANALYSIS - PCB-1248

	FACTOR						
Peak	pH	Temp.	Cl	pH x T	pH x Cl	T x Cl	pH x T x Cl
1	--*	--	--	(2,4),(7,24) (10,4)	except (7,2)	--	--
2	--	4	--	except (2,24)	except (2,0),(7,2)	--	--
3	--	--	--	--	--	--	--
4	--	--	--	--	--	U+	--
5	--	--	--	--	--	U	--
6	--	--	--	--	--	--	--
7	--	--	--	--	--	--	--

* Line indicates no significant difference.

+ U indicates results do not lend themselves to ready interpretation.

Mean Recoveries, %
(pH 10, 4°C)

Peak

1	2	3	4	5	6	7
92	94	93	93	92	92	92

Table A37

RECOMMENDED PRESERVATION CONDITIONS

Group	CONDITIONS		
	pH	Temperature, °C	Chlorine Effect
Single-compound pesticides	2	4	Marked for Aldrin
Chlordane	--*	4	None
Toxaphene	2-7	4	None
PCB-1254	--	4	None
PCB-1016	7	4	None
PCB-1260	2	4	None
PCB-1221	10	4	None
PCB-1232	10	4	None
PCB-1242	2	4	None
PCB-1248	10	4	None

* Line indicates no difference noted.

APPENDIX D

LIQUID-SOLID COLUMN CHROMATOGRAPHY TABLES

Table A38

FLORISIL - GROUP I

Mixed Pesticides

Fraction	Statistic	α-BHC	β-BHC	δ-BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
1	Mean	100.2	96.8	97.6	100.2	98.2	98.8	99.6	1.6
	St. Dev.	4.2	3.6	3.9	3.4	4.1	4.5	3.5	2.3
	Maximum	105	100	100	102	101	102	103	5
	Minimum	94	92	91	94	91	91	94	0
2	Mean	0.2	0.2	1.4	0.6	0.2	0.2	0.2	1.8
	St. Dev.	0.4	0.4	1.7	0.9	0.4	0.4	0.4	2.7
	Maximum	1	1	4	2	1	1	1	6
	Minimum	0	0	0	0	0	0	0	0
3	Mean	0.0	4.6	1.0	0.0	0.4	0.2	0.0	106.0
	St. Dev.	0.0	4.2	1.7	0.0	0.5	0.4	0.0	4.7
	Maximum	0	8	4	0	1	1	0	110
	Minimum	0	0	0	0	0	0	0	100
Total	Mean	100.4	101.6	100.0	100.6	98.6	99.2	99.8	109.4
	St. Dev.	4.2	2.6	4.1	3.4	4.2	4.7	3.7	1.3
	Maximum	105	106	104	104	101	103	103	111
	Minimum	94	100	93	95	91	91	94	108

Table A39

FLORISIL - GROUP II

Mixed Pesticides

Fraction	Statistic	γ-BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
1	Mean	100.4	100.0	100.2	37.0	0.2	3.6	0.0	0.0
	St. Dev.	4.0	4.6	3.7	10.0	0.4	1.8	0.0	0.0
	Maximum	105	105	105	46	1	6	0	0
	Minimum	96	96	96	20	0	1	0	0
2	Mean	0.8	0.8	0.6	63.8	101.0	96.4	6.8	68.4
	St. Dev.	1.1	0.8	0.5	9.4	6.0	6.3	3.1	10.2
	Maximum	2	2	1	79	110	105	10	82
	Minimum	0	0	0	56	94	88	3	58
3	Mean	0.0	0.4	0.2	0.6	0.2	0.0	91.2	25.6
	St. Dev.	0.0	0.5	0.4	0.5	0.4	0.0	4.1	7.6
	Maximum	0	1	1	1	1	0	96	35
	Minimum	0	0	0	0	0	0	86	18
Total	Mean	101.2	101.2	101.0	101.4	101.4	100.0	98.0	94.0
	St. Dev.	4.2	5.3	4.2	3.9	5.6	5.9	2.7	4.3
	Maximum	107	108	106	107	110	109	102	100
	Minimum	97	97	97	98	95	94	95	89

Table A40

FLORISIL - CHLORDANE

Fraction	Statistic	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6
1	Mean	101.8	100.8	101.8	101.2	99.8	97.6
	St. Dev.	2.3	1.9	2.2	1.8	1.1	9.8
	Maximum	105	103	104	103	101	113
	Minimum	99	98	99	99	98	87
2	Mean	1.2	1.0	1.0	1.8	1.8	1.2
	St. Dev.	1.1	1.2	2.2	2.0	2.0	1.6
	Maximum	3	3	5	5	5	4
	Minimum	0	0	0	0	0	0
3	Mean	0.0	0.2	0.0	0.0	0.0	0.0
	St. Dev.	0.0	0.4	0.0	0.0	0.0	0.0
	Maximum	0	1	0	0	0	0
	Minimum	0	0	0	0	0	0
Total	Mean	103.0	102.0	102.8	103.0	101.6	98.0
	St. Dev.	2.0	2.5	2.3	1.9	1.5	9.3
	Maximum	105	104	105	105	103	114
	Minimum	100	98	99	100	100	91

Table A41

FLORISIL - TOXAPHENE

Fraction	Statistic	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8
1	Mean	95.4	96.8	97.2	95.2	98.0	95.4	95.6	96.6
	St. Dev.	5.9	8.9	8.9	5.3	10.1	6.0	6.3	6.5
	Maximum	100	109	111	101	113	102	103	104
	Minimum	89	88	89	89	87	89	89	88
2	Mean	1.2	0.0	0.2	0.0	0.0	0.0	0.0	0.6
	St. Dev.	2.7	0.0	0.4	0.0	0.0	0.0	0.0	1.3
	Maximum	6	0	1	0	0	0	0	3
	Minimum	0	0	0	0	0	0	0	0
3	Mean	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	St. Dev.	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Maximum	0	0	0	0	0	0	0	0
	Minimum	0	0	0	0	0	0	0	0
Total	Mean	96.6	96.8	97.4	95.2	98.0	95.4	95.6	96.6
	St. Dev.	7.4	8.9	9.3	5.4	10.9	6.0	6.3	6.5
	Maximum	106	109	112	101	113	102	103	104
	Minimum	89	88	89	89	87	89	89	88

Table A42
FLORISIL - PCB-1254

		Peak						
Fraction	Statistic	1	2	3	4	5	6	7
1	Mean	90.6	90.8	90.8	90.4	90.4	90.0	90.4
	St. Dev.	5.1	5.1	6.5	5.4	5.4	5.4	5.4
	Maximum	97	97	98	97	97	97	97
	Minimum	84	84	82	83	85	83	83
2	Mean	0.8	0.8	0.8	0.8	0.8	0.8	0.8
	St. Dev.	1.8	1.8	1.8	1.8	1.8	1.8	1.8
	Maximum	4	4	4	4	4	4	4
	Minimum	0	0	0	0	0	0	0
3	Mean	0.0	0.0	0.0	0.0	1.0	0.0	0.0
	St. Dev.	0.0	0.0	0.0	0.0	2.2	0.0	0.0
	Maximum	0	0	0	0	5	0	0
	Minimum	0	0	0	0	0	0	0
Total	Mean	91.8	91.6	91.6	92.8	92.2	91.6	91.2
	St. Dev.	5.0	4.8	5.3	6.0	4.5	5.3	5.3
	Maximum	97	97	98	98	97	97	97
	Minimum	84	84	86	83	85	83	83

Table A43
FLORISIL - PCB-1016

		Peak							
Fraction	Statistic	1	2	3	4	5	6	7	8
1	Mean	98.0	97.0	97.6	96.4	97.8	97.6	96.8	95.6
	St. Dev.	2.1	1.9	1.5	1.5	2.0	1.5	1.9	2.1
	Maximum	101	99	99	99	101	99	99	99
	Minimum	95	94	95	95	96	95	94	94
2	Mean	1.4	0.6	0.6	0.0	0.0	0.0	0.0	0.0
	St. Dev.	1.1	0.5	0.5	0.0	0.0	0.0	0.0	0.0
	Maximum	3	1	1	0	0	0	0	0
	Minimum	0	0	0	0	0	0	0	0
3	Mean	2.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	St. Dev.	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Maximum	3	0	0	0	0	0	0	0
	Minimum	1	0	0	0	0	0	0	0
Total	Mean	102.2	97.6	98.2	96.4	97.8	97.6	96.8	95.6
	St. Dev.	1.3	2.2	1.9	1.5	2.0	1.5	1.9	2.1
	Maximum	104	100	100	99	101	99	99	99
	Minimum	101	94	95	95	96	95	94	94

Table A44

FLORISIL - PCB-1260

Fraction	Statistic	Peak 1	2	3	4	5	6	7	8	9	10	11
1	Mean	96.6	94.4	96.2	95.4	95.2	94.6	95.0	97.6	93.4	93.0	92.8
	St. Dev.	7.6	3.2	6.5	5.0	4.2	4.3	8.7	10.4	2.2	3.6	3.9
	Maximum	104	100	103	101	101	100	103	109	96	98	98
	Minimum	86	92	87	89	91	90	82	83	90	89	88
2	Mean	5.8	4.4	5.4	5.6	4.8	5.0	5.0	4.2	6.2	4.0	4.4
	St. Dev.	2.3	2.1	2.6	2.6	2.2	2.2	2.2	1.8	1.8	2.3	1.9
	Maximum	8	6	8	7	6	6	6	5	7	6	6
	Minimum	2	1	1	1	1	1	1	1	3	1	1
3	Mean	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	St. Dev.	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Maximum	0	0	0	0	0	0	0	0	0	0	0
	Minimum	0	0	0	0	0	0	0	0	0	0	0
Total	Mean	102.4	98.8	101.6	101.0	100.0	99.6	100.0	101.8	100.2	97.0	97.2
	St. Dev.	8.2	4.2	7.6	5.8	4.7	5.0	9.1	10.8	2.8	4.8	3.9
	Maximum	111	105	111	107	106	106	109	114	104	104	103
	Minimum	92	94	93	95	95	95	88	88	97	91	93

Table A45

FLORISIL - PCB-1221

Fraction	Statistic	Peak 1	2	3	4	5	6	7
1	Mean	97.2	96.8	96.8	97.0	97.8	97.0	96.8
	St. Dev.	1.6	1.8	3.6	4.8	2.4	2.8	4.1
	Maximum	99	99	100	101	101	100	100
	Minimum	95	95	91	89	95	94	90
2	Mean	0.4	0.0	0.0	0.2	1.0	0.0	0.0
	St. Dev.	0.9	0.0	0.0	0.4	0.7	0.0	0.0
	Maximum	2	0	0	1	2	0	0
	Minimum	0	0	0	0	0	0	0
3	Mean	0.2	0.0	0.0	0.4	0.6	0.0	0.0
	St. Dev.	0.4	0.0	0.0	0.9	0.5	0.0	0.0
	Maximum	1	0	0	2	1	0	0
	Minimum	0	0	0	0	0	0	0
Total	Mean	97.8	96.8	96.8	97.6	99.4	97.0	96.8
	St. Dev.	1.3	1.8	3.6	4.4	2.6	2.8	4.1
	Maximum	99	99	100	101	103	100	100
	Minimum	96	95	91	90	97	94	90

Table A46

FLORISIL - PCB-1232

Fraction	Statistic	Peak 1	2	3	4	5	6	7	8	9	10
1	Mean	92.6	94.6	94.0	93.8	94.6	99.2	101.2	93.4	93.8	96.2
	St. Dev.	5.4	5.1	4.4	4.5	5.5	5.1	5.4	4.7	5.9	7.2
	Maximum	101	99	99	98	98	108	106	99	101	107
	Minimum	88	86	89	87	85	96	94	86	86	88
2	Mean	4.2	3.4	5.4	5.8	7.2	5.0	1.6	7.0	3.4	1.4
	St. Dev.	2.3	2.5	2.7	2.8	2.9	3.2	3.6	7.3	4.8	3.1
	Maximum	8	7	10	10	10	8	8	19	10	7
	Minimum	2	0	3	3	3	0	0	0	0	0
3	Mean	0.0	1.8	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	St. Dev.	0.0	4.0	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Maximum	0	9	1	0	0	0	0	0	0	0
	Minimum	0	0	0	0	0	0	0	0	0	0
Total	Mean	96.8	99.8	99.8	99.6	101.8	104.0	102.8	100.4	97.2	97.6
	St. Dev.	6.1	2.8	4.5	3.6	4.2	6.0	3.7	4.2	6.0	7.1
	Maximum	105	102	104	103	106	108	106	105	105	107
	Minimum	90	95	94	94	95	99	97	95	90	88

Table A47

FLORISIL - PCB-1242

Fraction	Statistic	Peak 1	2	3	4	5	6	7	8	9
1	Mean	97.6	97.2	97.2	97.6	98.0	96.0	97.6	97.6	97.0
	St. Dev.	1.9	1.9	1.6	1.9	1.2	2.1	2.2	2.2	2.0
	Maximum	100	100	100	101	100	99	101	101	100
	Minimum	95	95	96	96	97	94	95	95	95
2	Mean	0.4	0.4	0.4	0.4	2.4	0.4	0.4	0.4	0.4
	St. Dev.	0.9	0.9	0.9	0.9	2.3	0.9	0.9	0.9	0.9
	Maximum	2	2	2	2	5	2	2	2	2
	Minimum	0	0	0	0	0	0	0	0	0
3	Mean	0.0	0.0	0.0	0.0	0.8	0.0	0.0	0.0	0.0
	St. Dev.	0.0	0.0	0.0	0.0	1.8	0.0	0.0	0.0	0.0
	Maximum	0	0	0	0	4	0	0	0	0
	Minimum	0	0	0	0	0	0	0	0	0
Total	Mean	98.0	97.6	97.6	98.0	101.6	96.4	98.0	98.0	97.2
	St. Dev.	2.0	1.8	1.5	1.7	4.5	1.8	2.2	1.7	1.6
	Maximum	100	100	100	101	109	99	101	101	100
	Minimum	95	95	96	97	97	94	95	97	96

Table A48

FLORISIL - PCB-1248

Fraction	Statistic	Peak						
		1	2	3	4	5	6	7
1	Mean	102.2	103.8	102.8	104.0	104.4	103.8	103.0
	St. Dev.	4.3	5.8	5.8	4.0	3.0	7.0	6.9
	Maximum	109.0	113.0	112.0	110.0	109.0	116.0	114.0
	Minimum	97.0	97.0	96.0	99.0	101.0	98.0	95.0
2	Mean	0.2	0.2	0.0	0.4	0.0	0.2	0.0
	St. Dev.	0.4	0.4	0.0	0.5	0.0	0.4	0.0
	Maximum	1	1	0	1	0	1	0
	Minimum	0	0	0	0	0	0	0
3	Mean	0.0	0.4	0.0	0.2	0.4	0.0	0.0
	St. Dev.	0.0	0.5	0.0	0.4	0.9	0.0	0.0
	Maximum	0	1	0	1	2	0	0
	Minimum	0	0	0	0	0	0	0
Total	Mean	102.4	104.4	102.8	104.6	104.8	104.0	103.0
	St. Dev.	4.3	6.1	5.8	3.9	3.8	6.9	6.9
	Maximum	109	114	112	110	111	116	114
	Minimum	97	97	96	100	101	99	95

Table A49

ALUMINA - GROUP I

Mixed Pesticides

Fraction	Statistic	α-BHC	β-BHC	δ-BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
1	Mean	26.8	0.4	0.0	0.0	90.6	16.4	82.0	0.0
	St. Dev.	5.3	0.5	0.0	0.0	11.3	5.3	11.4	0.0
	Maximum	34	1	0	0	103	24	91	0
	Minimum	20	0	0	0	72	10	62	0
2	Mean	69.4	14.8	0.4	98.6	5.8	83.4	10.8	0.0
	St. Dev.	4.0	11.3	0.5	3.0	11.8	3.4	12.2	0.0
	Maximum	75	31	1	102	27	88	32	0
	Minimum	64	5	0	94	0	80	1	0
3	Mean	0.2	87.2	97.8	0.0	0.0	0.0	0.0	101.8
	St. Dev.	0.4	9.1	2.5	0.0	0.0	0.0	0.0	3.4
	Maximum	1	95	101	0	0	0	0	106
	Minimum	0	75	96	0	0	0	0	99
Total	Mean	95.4	102.4	98.2	98.6	96.4	99.8	92.8	101.8
	St. Dev.	1.7	3.2	3.0	3.0	5.1	4.1	2.3	3.4
	Maximum	98	107	102	102	104	104	96	106
	Minimum	94	99	96	94	92	95	90	99

Table A50

ALUMINA - GROUP II

Mixed Pesticides

Fraction	Statistic	γ-BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
1	Mean	1.6	93.4	97.0	0.6	0.0	0.0	0.0	0.0
	St. Dev.	1.3	5.6	5.8	0.5	0.0	0.0	0.0	0.0
	Maximum	3	102	103	1	0	0	0	0
	Minimum	0	88	90	0	0	0	0	0
2	Mean	103.2	6.4	1.6	91.4	104.4	105.0	0.0	0.8
	St. Dev.	5.7	3.6	1.3	7.2	6.1	5.7	0.0	0.4
	Maximum	112	9	3	103	114	115	0	1
	Minimum	97	2	0	84	97	101	0	0
3	Mean	0.2	0.4	0.0	2.8	3.0	0.0	85.6	104.6
	St. Dev.	0.4	0.5	0.0	0.8	2.5	0.0	2.5	6.0
	Maximum	1	1	0	4	7	0	88	112
	Minimum	0	0	0	2	0	0	83	99
Total	Mean	105.0	100.2	98.6	94.8	107.4	105.0	85.6	105.4
	St. Dev.	5.5	5.4	5.2	7.7	5.6	5.7	2.5	6.2
	Maximum	114	105	104	107	117	115	88	113
	Minimum	100	92	91	87	103	101	83	99

Table A51

ALUMINA - CHLORDANE

Fraction	Statistic	Peak					
		1	2	3	4	5	6
1	Mean	97.0	94.2	94.8	61.0	55.0	80.8
	St. Dev.	5.1	4.2	5.1	24.5	15.4	7.0
	Maximum	103	97	99	86	71	89
	Minimum	90	87	86	30	33	72
2	Mean	3.6	3.0	4.8	40.4	40.8	7.6
	St. Dev.	2.6	4.0	4.0	25.2	15.4	6.9
	Maximum	7	10	10	74	61	13
	Minimum	1	0	0	16	24	0
3	Mean	0.0	0.8	0.6	0.0	0.8	5.8
	St. Dev.	0.0	0.4	0.5	0.0	0.4	0.4
	Maximum	0	1	1	0	1	6
	Minimum	0	0	0	0	0	5
Total	Mean	100.6	98.0	100.2	101.4	94.6	94.2
	St. Dev.	2.7	1.2	3.9	3.3	2.9	3.8
	Maximum	104	100	104	104	98	100
	Minimum	97	97	96	96	90	90

Table A52
ALUMINA - TOXAPHENE

Fraction	Statistic	Peak 1	2	3	4	5	6	7	8
	Mean	78.6	75.8	63.4	59.0	66.6	63.6	62.6	64.2
1	St. Dev.	4.3	3.1	5.0	9.4	3.3	5.3	6.3	3.6
	Maximum	84	81	70	68	72	70	71	70
	Minimum	72	73	57	47	63	56	55	60
	Mean	22.4	24.2	31.6	38.4	22.0	36.6	34.4	22.8
2	St. Dev.	4.0	2.4	4.6	9.2	2.1	6.3	7.0	2.7
	Maximum	28	27	37	49	24	44	42	26
	Minimum	19	21	27	29	19	30	27	20
	Mean	0.0	0.0	0.0	0.0	0.0	0.0	0.0	5.8
3	St. Dev.	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.4
	Maximum	0	0	0	0	0	0	0	6
	Minimum	0	0	0	0	0	0	0	5
	Mean	101.0	100.0	95.0	97.4	88.6	100.2	97.0	92.8
Total	St. Dev.	2.4	2.1	2.1	2.4	2.1	3.1	3.5	4.0
	Maximum	104	102	97	100	91	103	99	96
	Minimum	98	97	92	94	86	95	91	86

Table A53
ALUMINA - PCB-1254

Fraction	Statistic	Peak 1	2	3	4	5	6	7
	Mean	100.6	100.6	100.2	101.8	100.4	100.2	98.4
1	St. Dev.	1.8	1.5	1.9	1.6	4.5	1.6	4.4
	Maximum	103	103	103	104	108	103	103
	Minimum	99	99	98	100	97	99	91
	Mean	0.4	1.0	0.6	1.0	3.2	1.0	0.4
2	St. Dev.	0.5	0.0	0.9	0.0	1.3	0.0	0.5
	Maximum	1	1	2	1	5	1	1
	Minimum	0	1	0	1	2	1	0
	Mean	0.0	0.0	1.6	0.0	0.0	0.0	0.0
3	St. Dev.	0.0	0.0	0.5	0.0	0.0	0.0	0.0
	Maximum	0	0	2	0	0	0	0
	Minimum	0	0	1	0	0	0	0
	Mean	101.0	101.6	102.4	102.8	103.6	101.2	98.8
Total	St. Dev.	2.0	1.5	1.8	1.6	3.9	1.6	4.3
	Maximum	104	104	105	105	110	104	104
	Minimum	99	100	100	101	100	100	92

Table A54

ALUMINA - PCB-1016

Fraction	Statistic	Peak 1	 2	 3	 4	 5	 6	 7	 8
1	Mean	98.6	98.8	98.6	98.0	98.6	98.6	99.0	98.8
	St. Dev.	1.3	1.3	1.1	2.4	1.5	1.5	1.2	1.6
	Maximum	100	100	100	101	100	100	100	100
	Minimum	97	97	97	95	97	97	97	97
2	Mean	0.4	0.8	1.0	0.2	0.0	0.0	0.6	0.0
	St. Dev.	0.5	0.4	0.0	0.4	0.0	0.0	0.9	0.0
	Maximum	1	1	1	1	0	0	2	0
	Minimum	0	0	1	0	0	0	0	0
3	Mean	0.0	0.0	0.2	0.0	0.0	0.0	0.0	0.0
	St. Dev.	0.0	0.0	0.4	0.0	0.0	0.0	0.0	0.0
	Maximum	0	0	1	0	0	0	0	0
	Minimum	0	0	0	0	0	0	0	0
Total	Mean	99.0	99.6	99.8	98.2	98.6	98.6	99.6	98.8
	St. Dev.	1.6	1.1	1.5	2.3	1.5	1.5	1.5	1.6
	Maximum	101	101	102	101	100	100	101	100
	Minimum	97	98	98	95	97	97	97	97

Table A55

ALUMINA - PCB-1260

Fraction	Statistic	Peak 1	 2	 3	 4	 5	 6	 7	 8	 9	 10	 11
1	Mean	100.0	99.6	99.6	100.0	99.6	100.0	100.2	99.4	100.2	99.8	100.2
	St. Dev.	3.7	3.5	2.9	3.1	3.7	3.1	3.3	3.3	3.3	3.0	3.3
	Maximum	104	103	102	103	103	103	103	103	104	104	103
	Minimum	95	94	95	95	94	95	95	94	95	94	95
2	Mean	2.0	1.8	2.0	2.6	2.2	2.0	2.0	2.0	2.6	2.0	1.4
	St. Dev.	1.9	1.6	1.9	2.2	1.6	1.9	1.9	1.9	2.3	1.9	1.5
	Maximum	4	4	4	5	4	4	4	4	5	4	3
	Minimum	0	0	0	1	1	0	0	0	0	0	0
3	Mean	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.2	0.0	0.0
	St. Dev.	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.8	0.0	0.0
	Maximum	0	0	0	0	0	0	0	0	2	0	0
	Minimum	0	0	0	0	0	0	0	0	0	0	0
Total	Mean	102.0	101.4	101.6	102.6	101.8	102.0	102.2	101.4	104.0	101.8	101.6
	St. Dev.	4.7	4.6	4.1	4.3	4.5	4.5	4.5	4.3	4.5	5.1	4.1
	Maximum	108	106	106	107	107	107	107	104	108	107	106
	Minimum	95	94	95	96	95	95	95	94	97	94	95

Table A56

ALUMINA - PCB-1221

Fraction	Statistic	Peak						
		1	2	3	4	5	6	7
1	Mean	98.0	97.2	99.8	99.8	98.8	99.0	99.2
	St. Dev.	2.9	2.4	1.3	1.3	1.5	1.9	2.4
	Maximum	102	101	101	101	101	102	103
	Minimum	95	95	98	98	97	97	97
2	Mean	0.8	0.0	0.4	0.4	0.8	0.0	0.6
	St. Dev.	0.4	0.0	0.5	0.5	0.4	0.0	0.5
	Maximum	1	0	1	1	1	0	1
	Minimum	0	0	0	0	0	0	0
3	Mean	1.6	0.0	0.0	0.0	0.0	0.4	0.0
	St. Dev.	0.5	0.0	0.0	0.0	0.0	0.5	0.0
	Maximum	2	0	0	0	0	1	0
	Minimum	1	0	0	0	0	0	0
Total	Mean	100.4	97.2	100.2	100.2	99.6	99.4	99.8
	St. Dev.	3.1	2.4	1.8	1.3	1.1	1.8	2.2
	Maximum	105	101	102	102	101	102	103
	Minimum	97	95	98	99	98	97	98

Table A57

ALUMINA - PCB-1232

Fraction	Statistic	Peak									
		1	2	3	4	5	6	7	8	9	10
1	Mean	98.8	98.0	97.8	98.0	98.4	99.2	100.4	98.0	99.4	96.8
	St. Dev.	2.8	2.8	2.6	2.2	2.6	3.0	3.8	2.7	4.3	3.6
	Maximum	101	102	101	101	102	104	106	101	105	102
	Minimum	94	94	94	95	95	96	96	94	95	92
2	Mean	1.2	0.2	1.0	0.2	0.8	0.4	0.0	0.0	0.0	0.0
	St. Dev.	0.8	0.4	0.7	0.4	0.4	0.9	0.0	0.0	0.0	0.0
	Maximum	2	1	2	1	1	2	0	0	0	0
	Minimum	0	0	0	0	0	0	0	0	0	0
3	Mean	0.0	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	St. Dev.	0.0	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Maximum	0	1	0	0	0	0	0	0	0	0
	Minimum	0	0	0	0	0	0	0	0	0	0
Total	Mean	100.0	98.4	98.8	98.2	99.2	99.6	100.4	98.0	99.4	96.8
	St. Dev.	3.0	3.0	2.3	2.3	2.3	3.3	3.8	2.7	4.3	3.6
	Maximum	103	102	101	101	102	104	106	101	105	102
	Minimum	95	94	95	95	96	96	96	94	95	92

Table A58

ALUMINA - PCB-1242

Fraction	Statistic	Peak 1	2	3	4	5	6	7	8	9
	Mean	99.6	99.4	98.4	97.2	97.8	97.8	98.0	98.0	98.0
1	St. Dev.	3.1	2.3	2.5	2.2	3.6	1.6	2.5	1.4	1.9
	Maximum	105	103	102	100	101	99	101	100	100
	Minimum	97	97	95	94	92	95	94	96	95
	Mean	1.6	1.4	1.0	0.4	0.2	0.2	0.2	0.0	0.0
2	St. Dev.	0.5	0.5	0.0	0.9	0.4	0.4	0.4	0.0	0.0
	Maximum	2	2	1	2	1	1	1	0	0
	Minimum	1	1	1	0	0	0	0	0	0
	Mean	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3	St. Dev.	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Maximum	1	0	0	0	0	0	0	0	0
	Minimum	0	0	0	0	0	0	0	0	0
	Mean	101.6	100.8	99.4	97.6	98.0	98.0	98.2	98.0	98.0
Total	St. Dev.	3.6	2.0	2.5	2.3	3.5	1.7	2.6	1.4	1.9
	Maximum	108	104	103	100	101	99	101	100	100
	Minimum	99	99	96	94	92	95	94	96	95

Table A59

ALUMINA - PCB-1248

Fraction	Statistic	Peak 1	2	3	4	5	6	7
	Mean	98.4	98.8	98.0	98.2	97.8	98.0	97.6
1	St. Dev.	1.3	1.1	1.4	1.1	1.3	2.4	1.7
	Maximum	100	100	99	99	99	101	100
	Minimum	97	97	96	97	96	95	96
	Mean	2.0	1.4	1.4	2.2	1.8	2.0	2.0
2	St. Dev.	1.7	0.9	0.9	1.6	1.8	1.2	1.7
	Maximum	5	3	3	5	5	4	5
	Minimum	1	1	1	1	1	1	1
	Mean	0.0	0.0	0.0	0.2	0.0	0.0	0.0
3	St. Dev.	0.0	0.0	0.0	0.4	0.0	0.0	0.0
	Maximum	0	0	0	1	0	0	0
	Minimum	0	0	0	0	0	0	0
	Mean	100.4	100.2	99.4	100.6	99.6	100.0	99.6
Total	St. Dev.	2.3	1.8	2.2	2.2	2.3	3.3	3.1
	Maximum	104	103	102	104	103	105	105
	Minimum	98	98	97	98	97	97	97

APPENDIX E

WASTEWATER APPLICATION FIGURES AND DATA TABLES

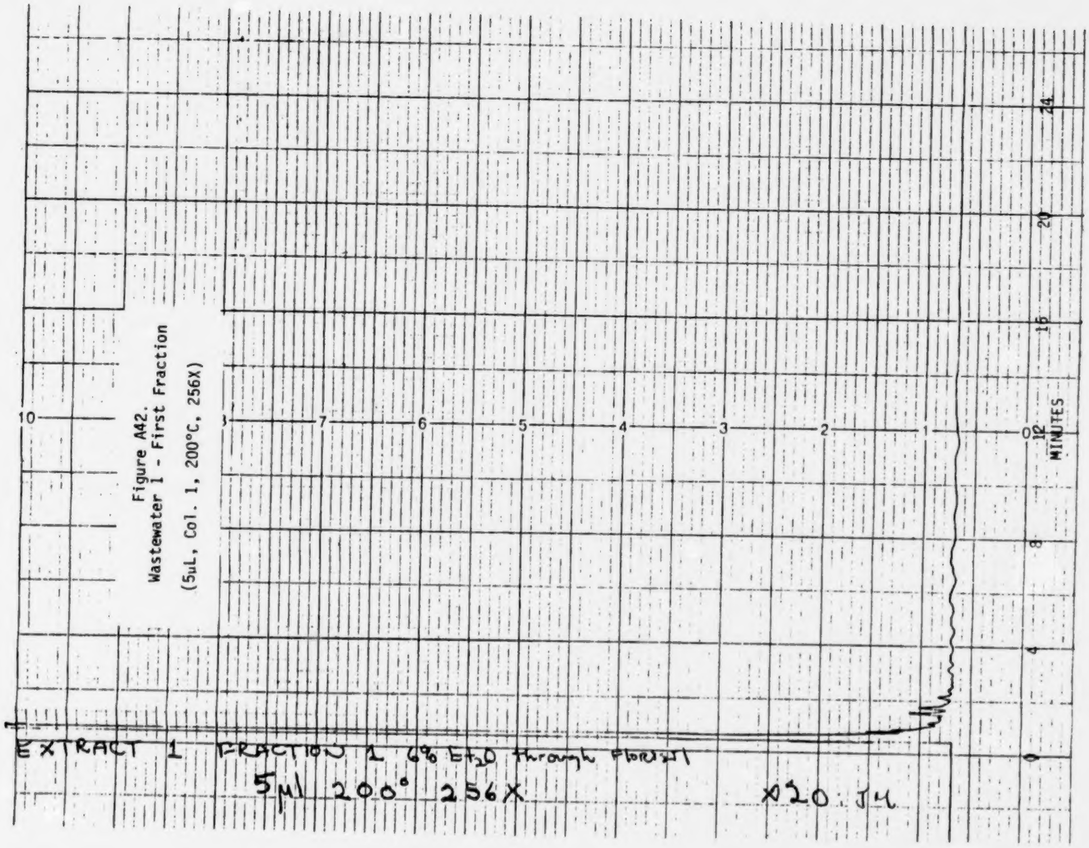


Figure A42.
Wastewater I - First Fraction
(5uL, Col. 1, 200°C, 256X)

EXTRACT 1 FRACTION 1 6% Et_2O through Florisil
5µl 200° 256X ×20 JM

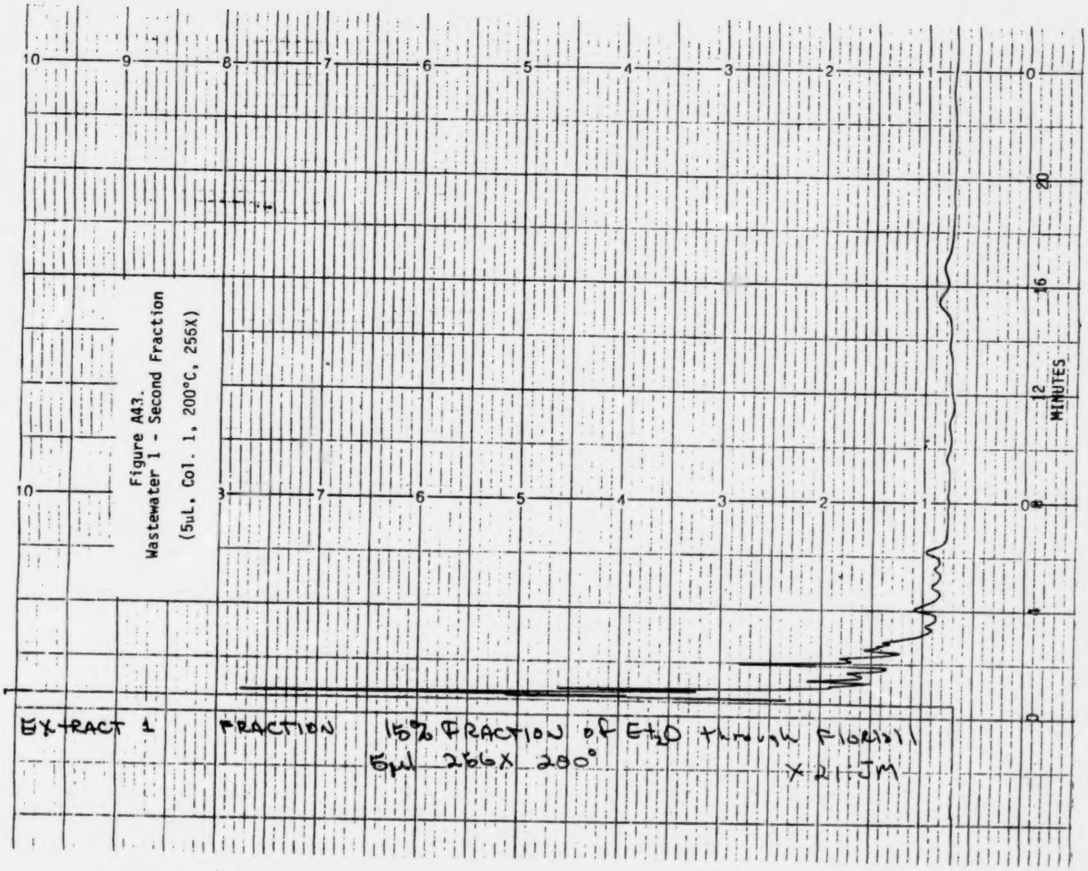
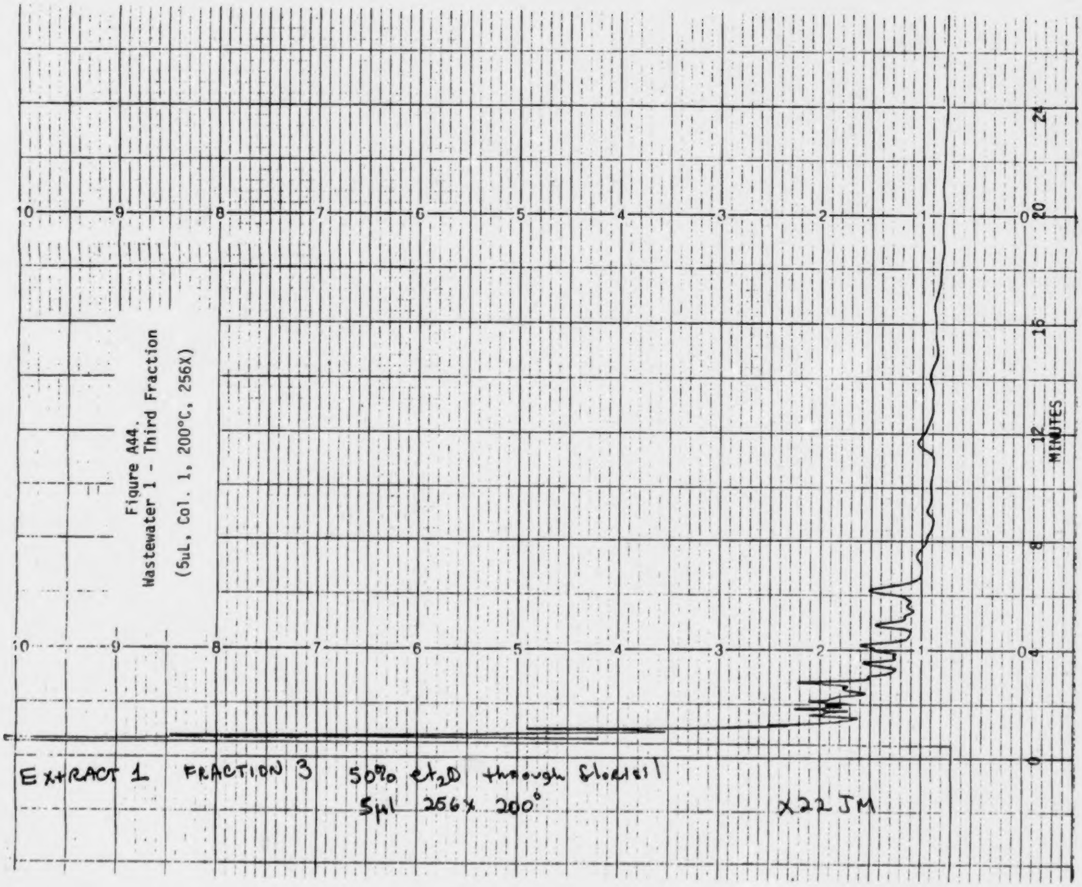


Figure A43.
Wastewater I - Second Fraction
(5uL, Col. 1, 200°C, 256X)

EXTRACT 1 FRACTION 15% FRACTION of Et_2O through Florisil
5µl 256X 200°
X 21 JM

Figure A44.
Wastewater 1 - Third Fraction
(5uL, Col. 1, 200°C, 256X)



EXTRACT 1 FRACTION 3 50% et$_2$O through florisil
5μl 256x 200°
X22 JM

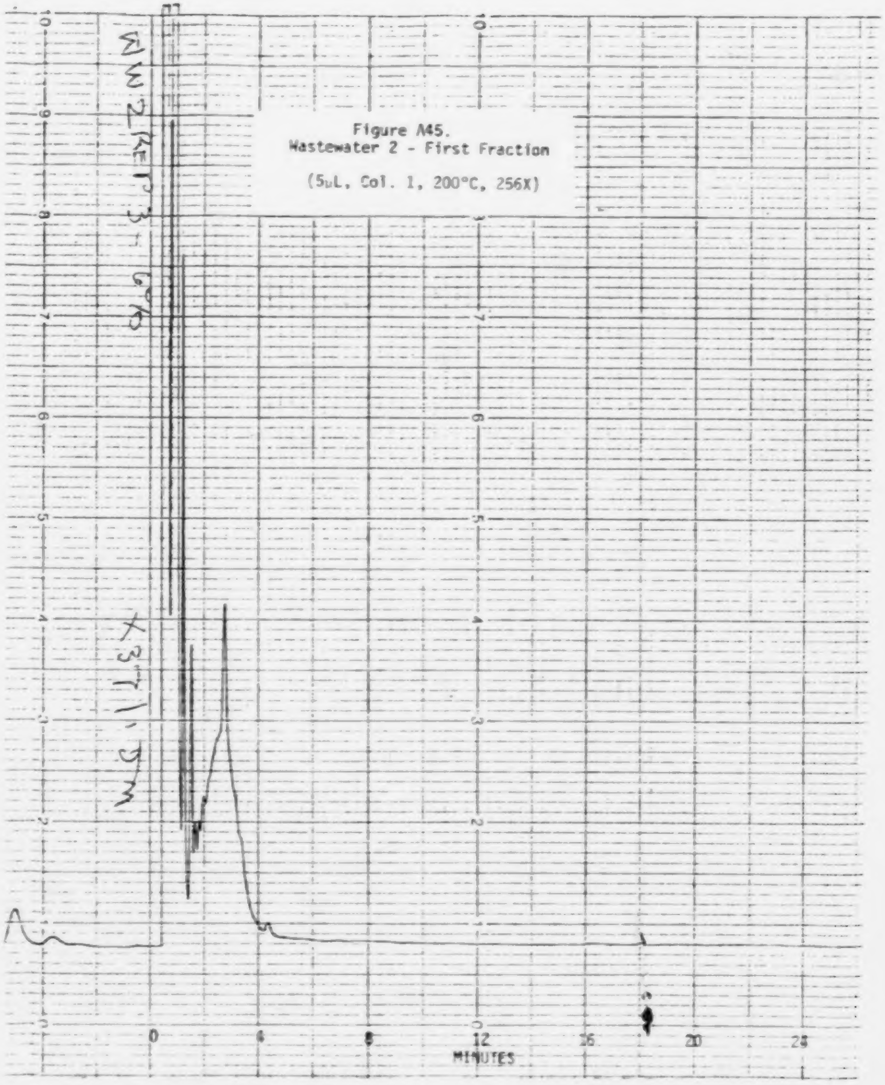


Figure A45.
Wastewater 2 - First Fraction

(5uL, Col. 1, 200°C, 256X)

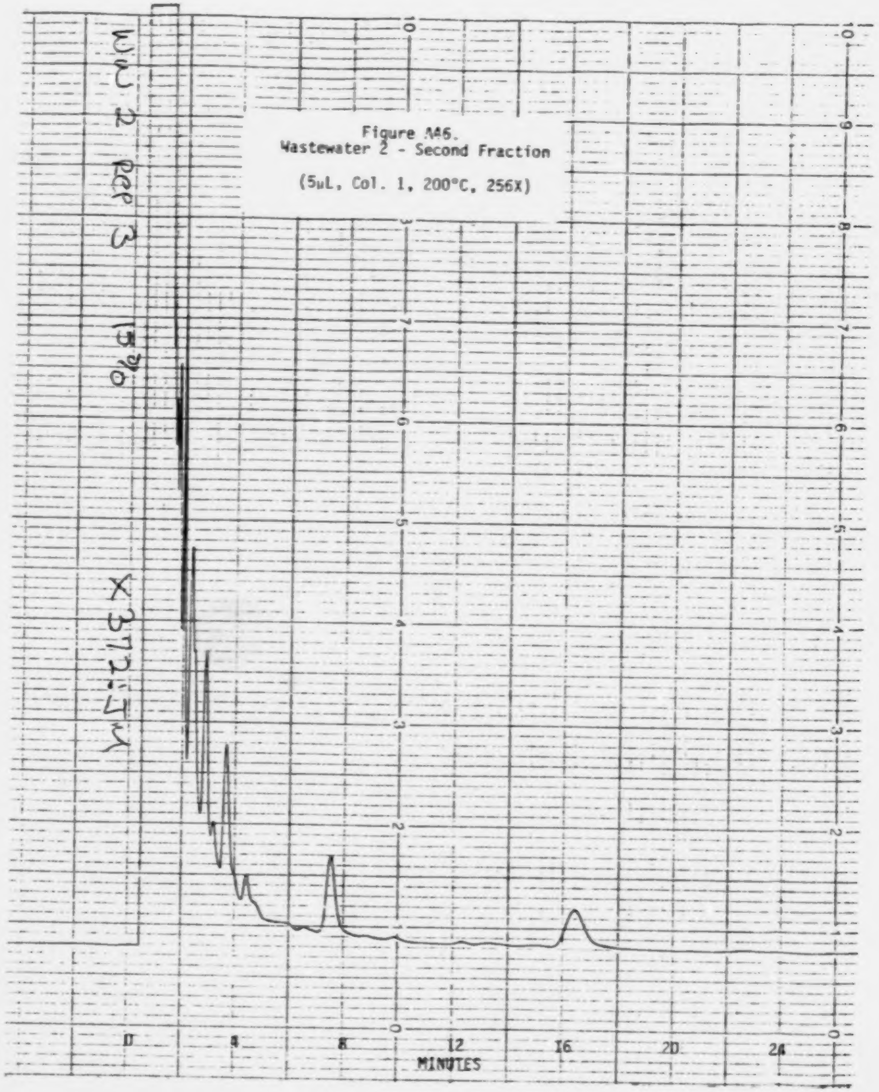


Figure A46.
Wastewater 2 - Second Fraction

(5µL, Col. 1, 200°C, 256X)

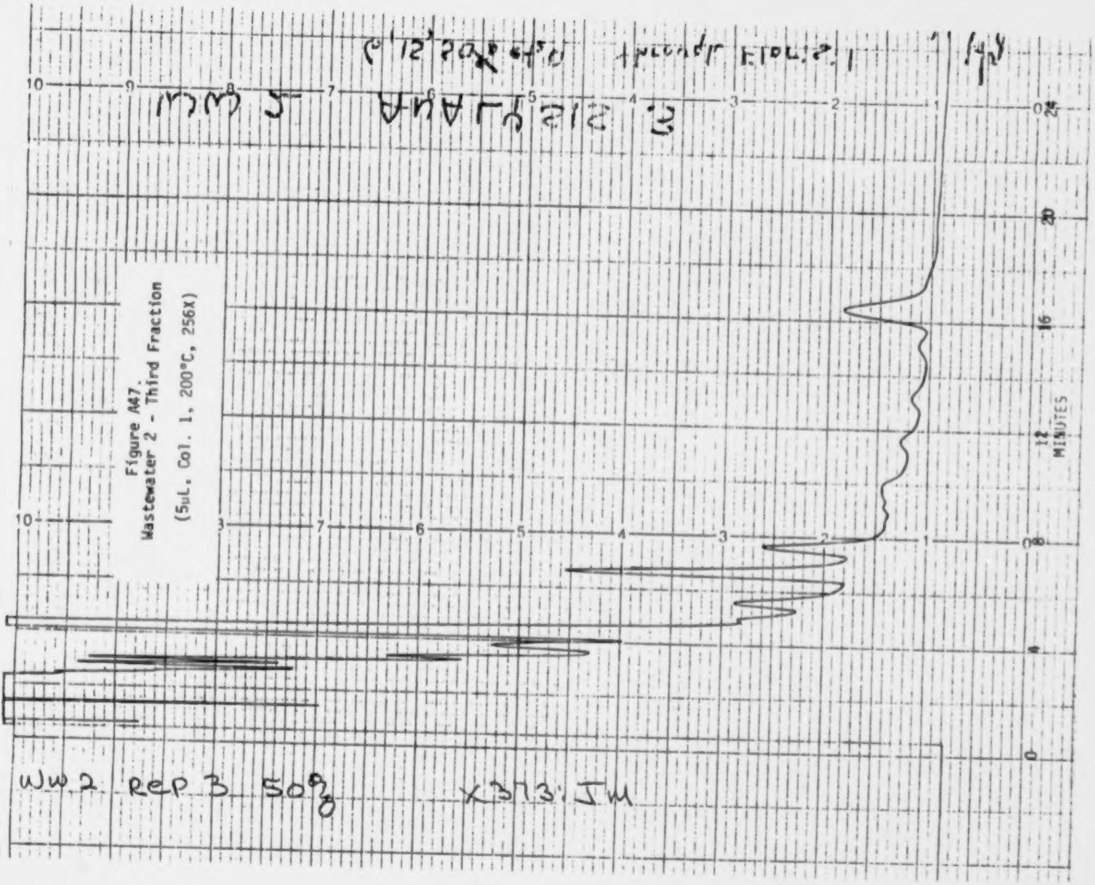


Figure A47.
Wastewater 2 - Third Fraction
(5uL, Col. 1, 200°C, 256X)

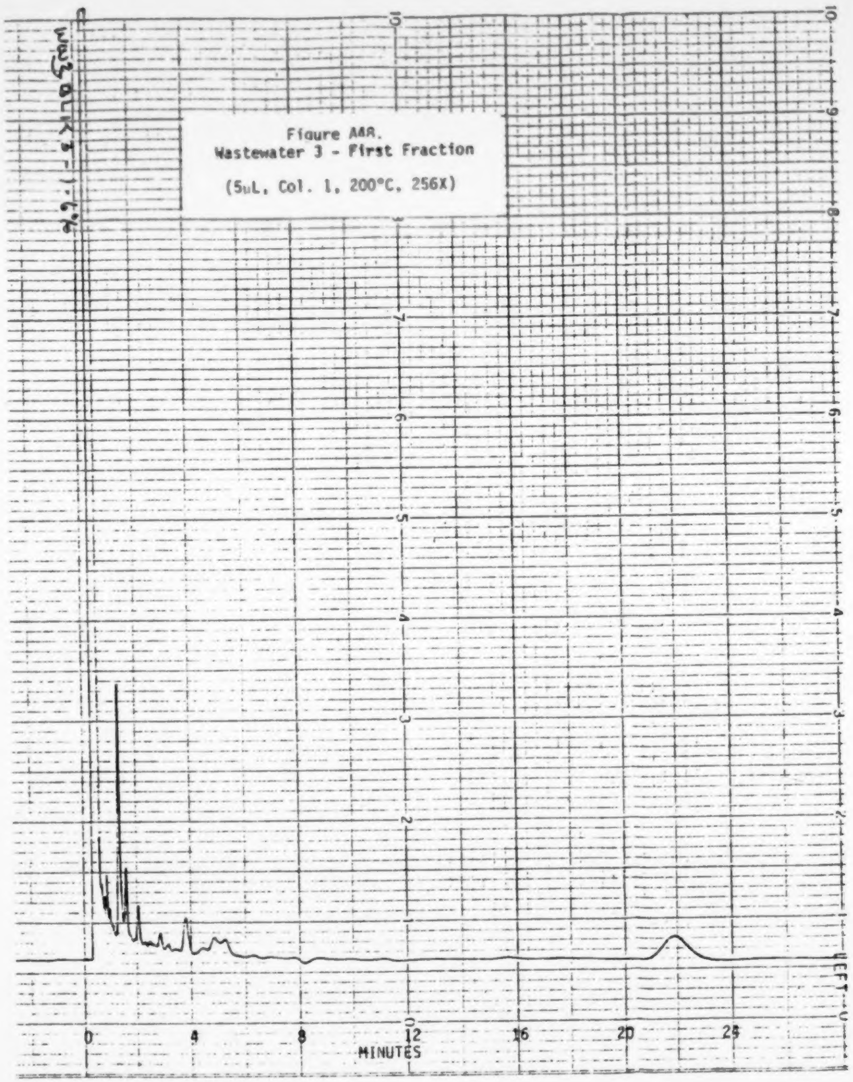


Figure A48.
Wastewater 3 - First Fraction

(5uL, Col. 1, 200°C, 256X)

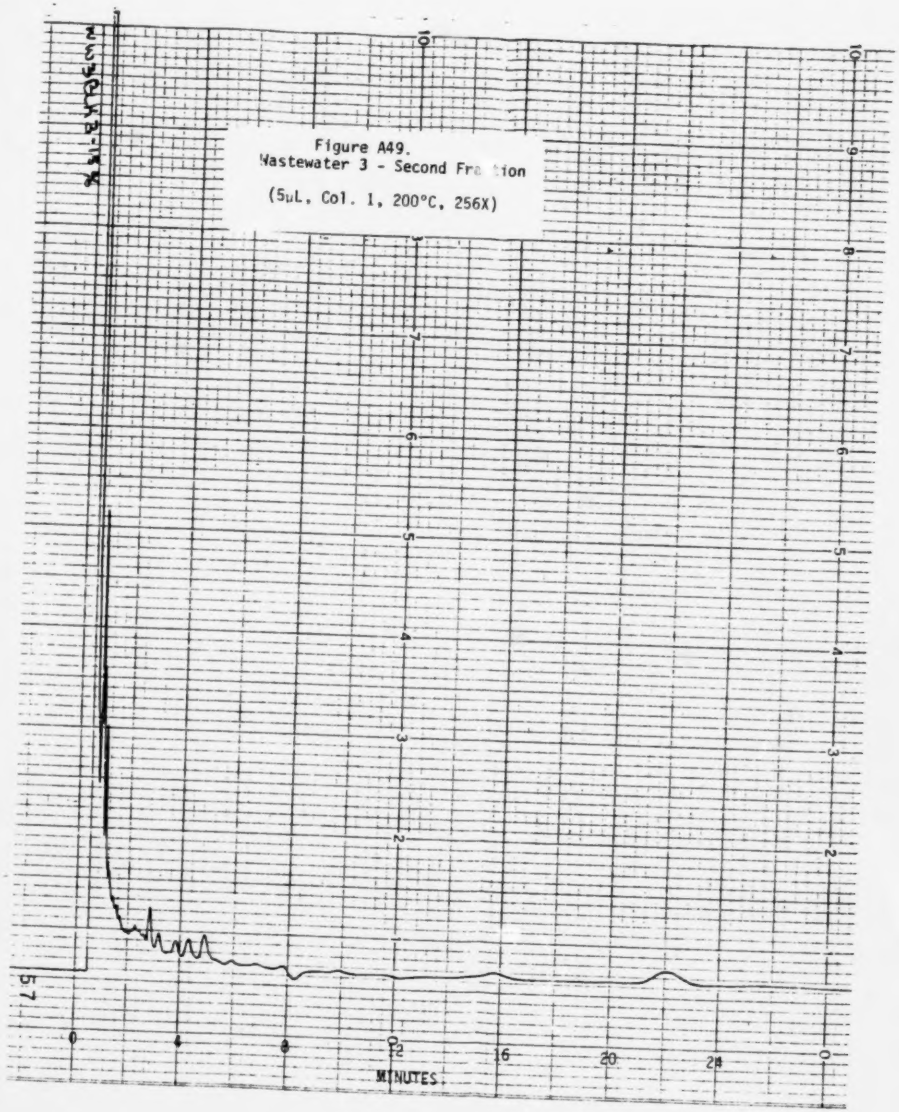


Figure A49.
Wastewater 3 - Second Fraction

(5μL, Col. 1, 200°C, 256X)

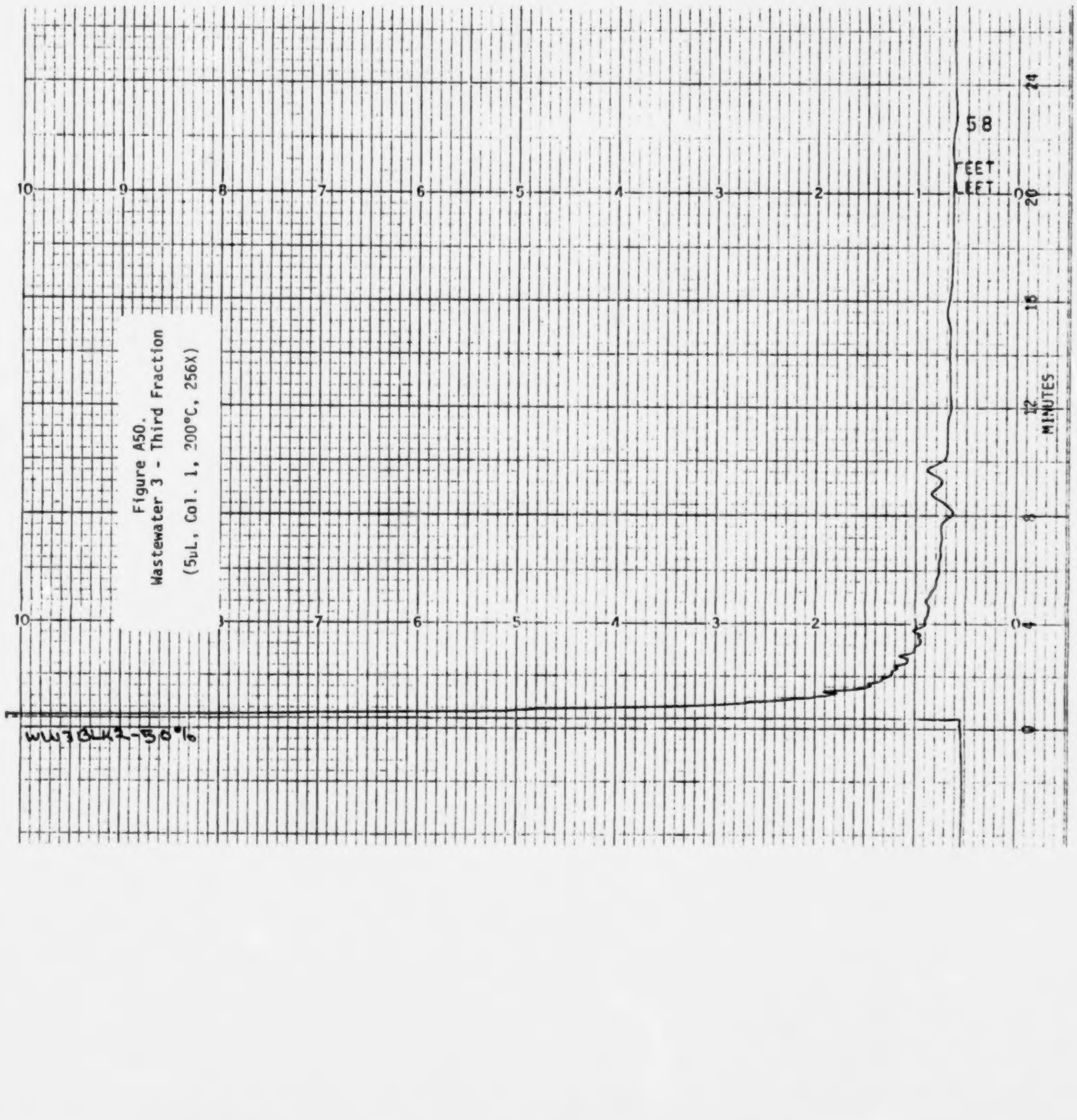


Figure A50.
Wastewater 3 - Third Fraction
(5µL, Col. 1, 200°C, 256X)

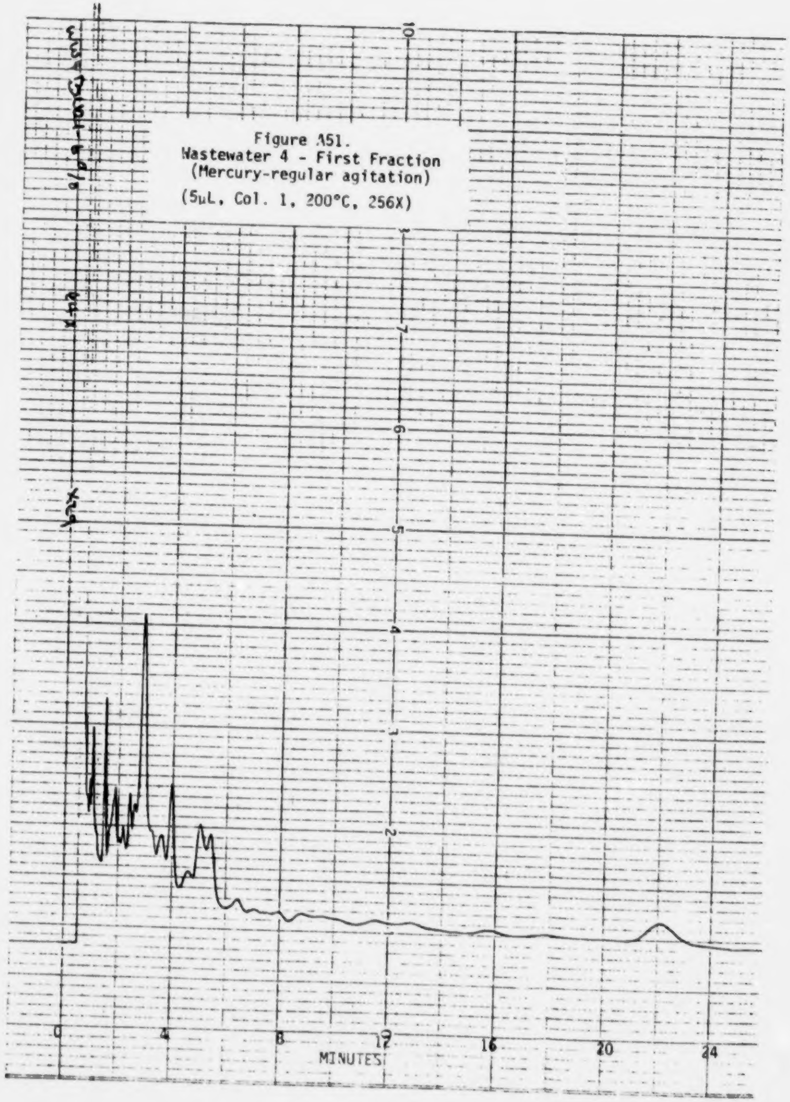


Figure 151.
Wastewater 4 - First Fraction
(Mercury-regular agitation)
(5μL, Col. 1, 200°C, 256X)

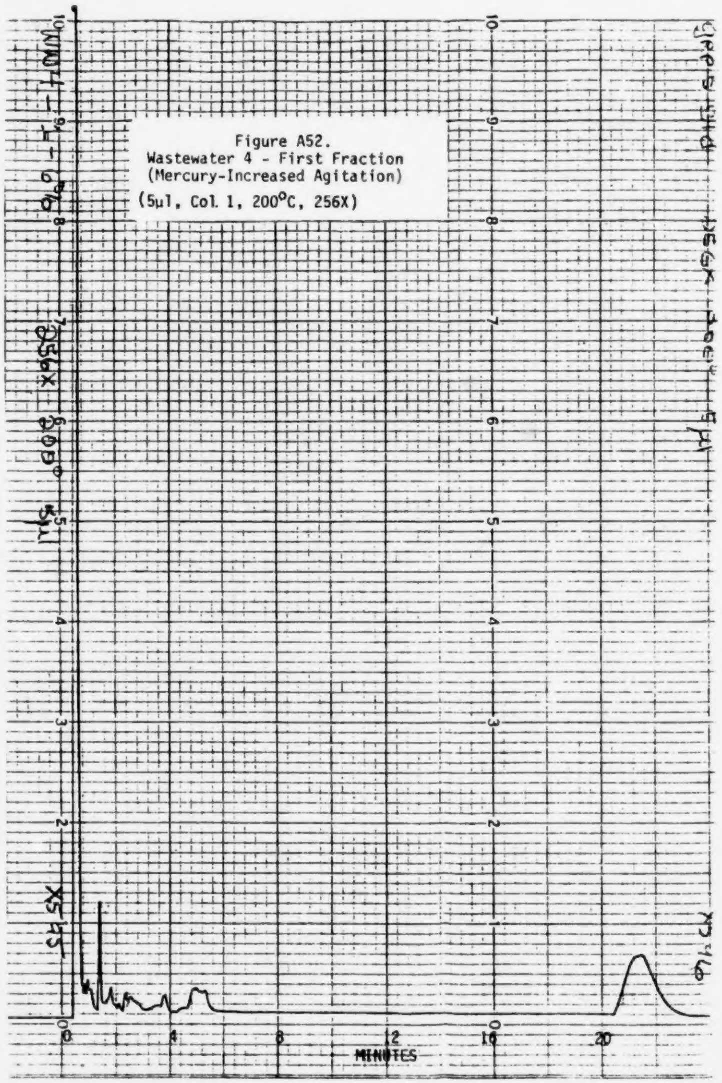


Figure A52.
Wastewater 4 - First Fraction
(Mercury-Increased Agitation)
(5µl, Col. 1, 200°C, 256X)

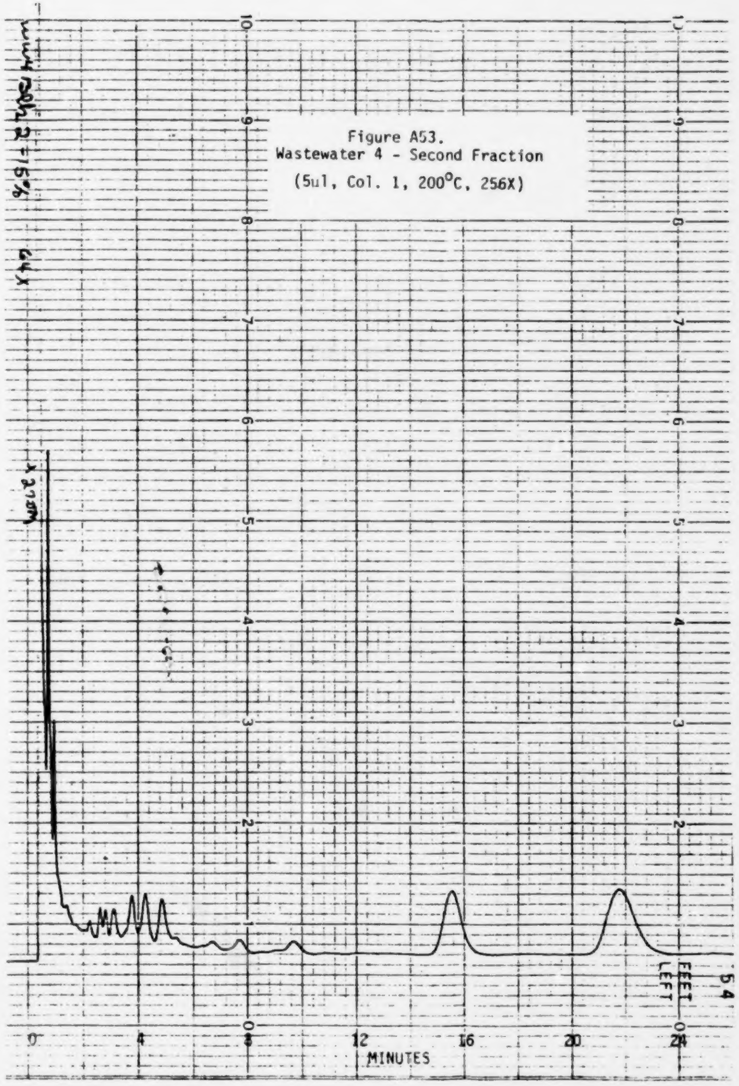


Figure A53.
Wastewater 4 - Second Fraction
(5µl, Col. 1, 200°C, 256X)

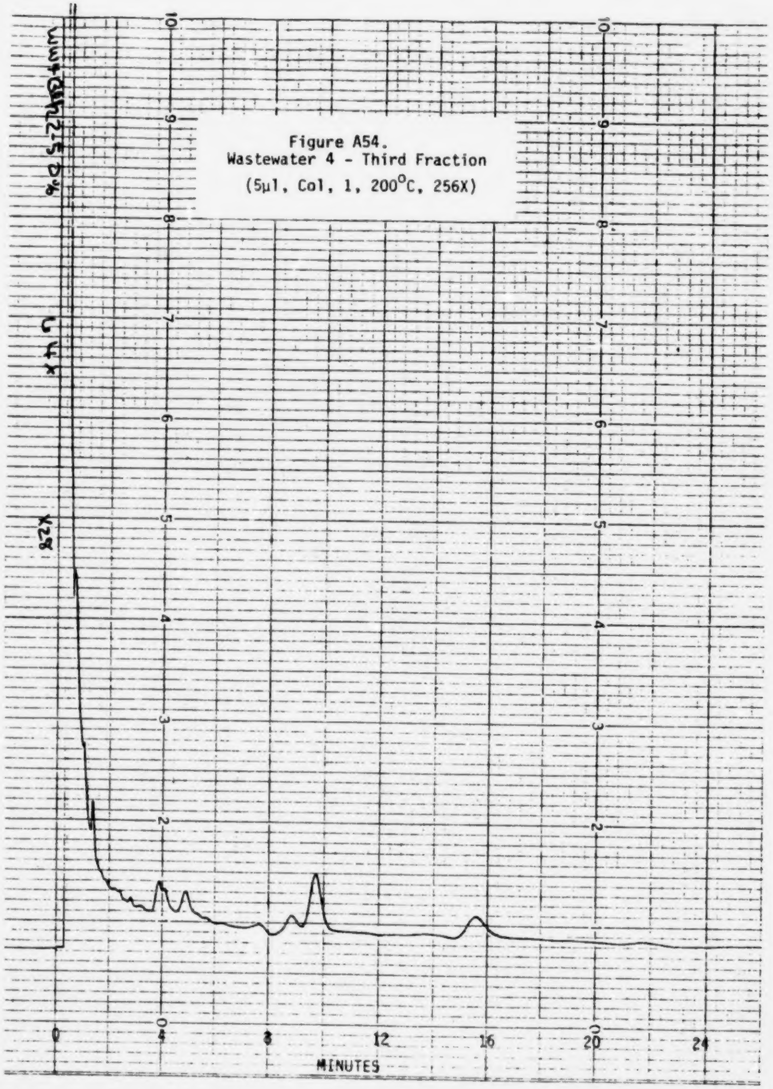


Figure A54.
Wastewater 4 - Third Fraction
(5µl, Col, 1, 200°C, 256X)

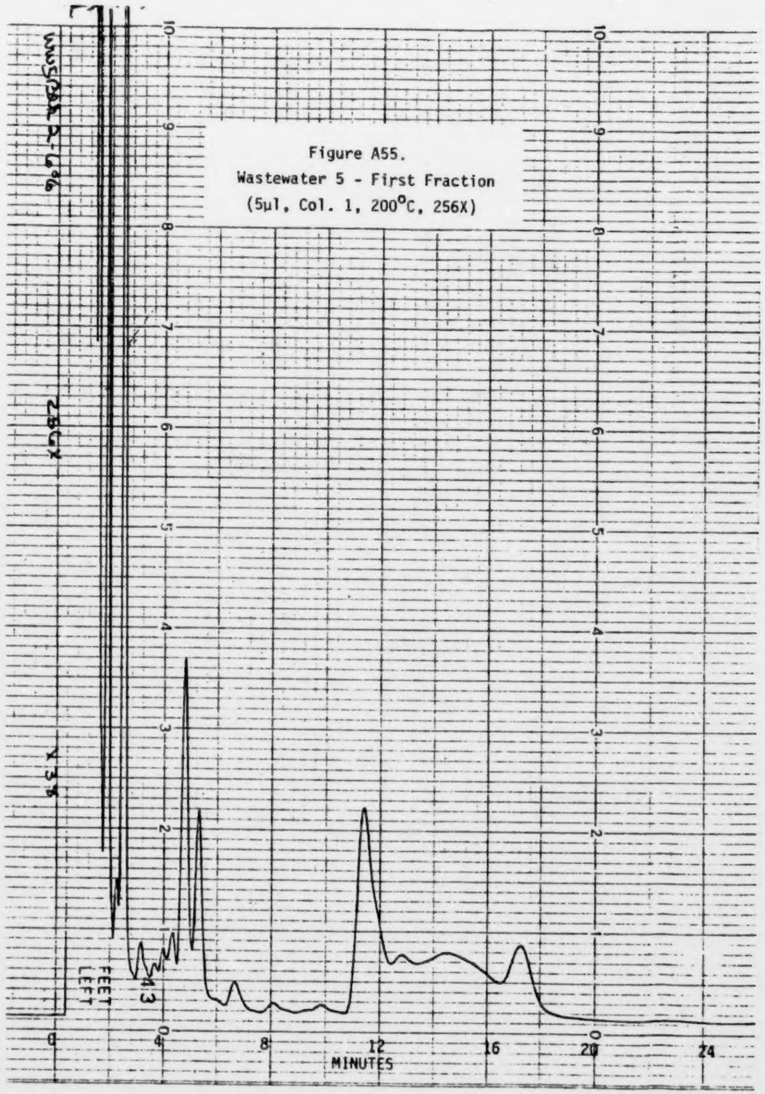


Figure A55.
Wastewater 5 - First Fraction
(5μl, Col. 1, 200°C, 256X)

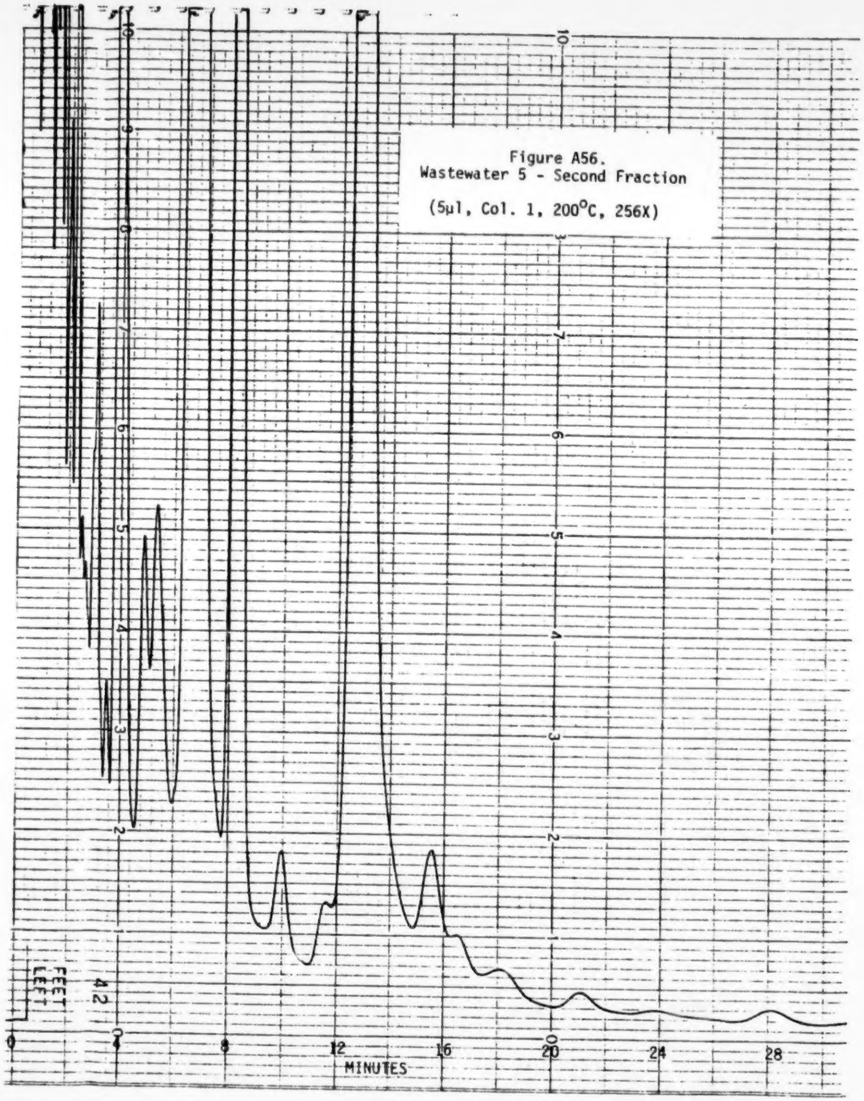


Figure A56.
Wastewater 5 - Second Fraction

(5μl, Col. 1, 200°C, 256X)

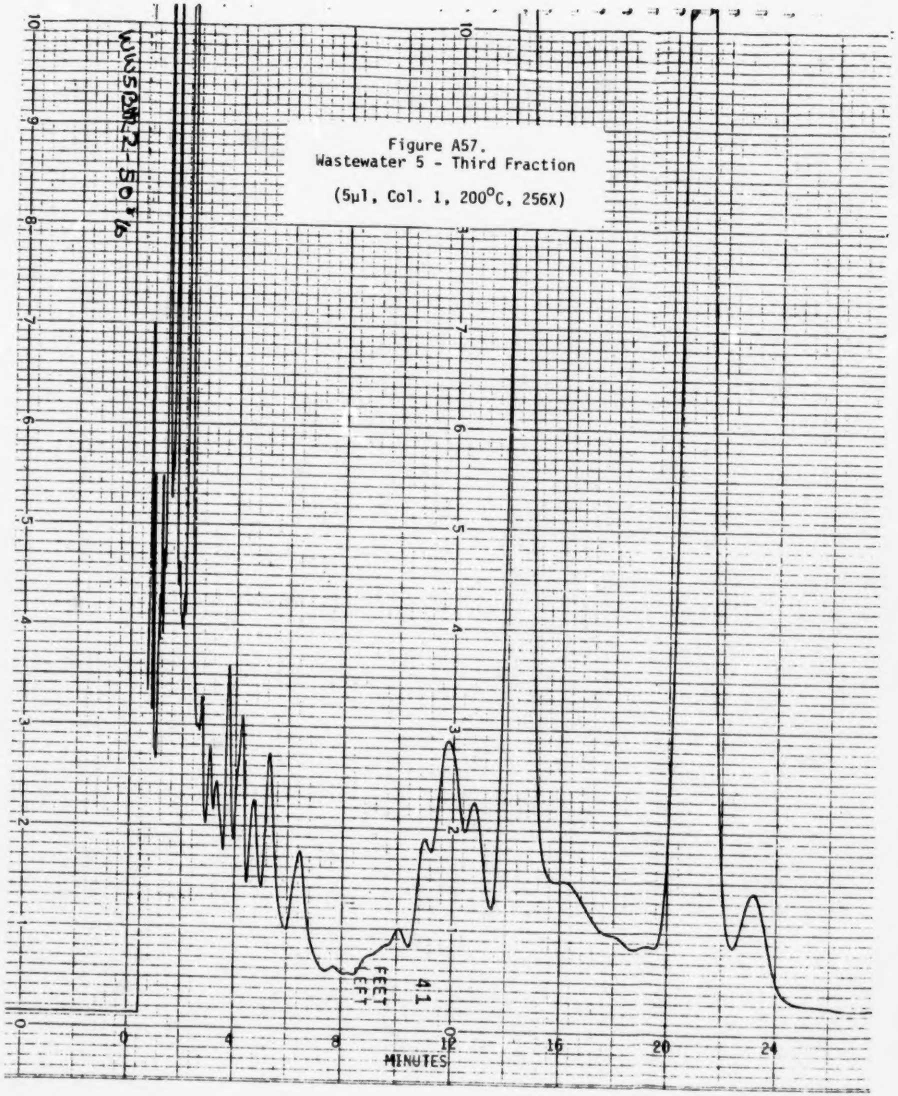


Figure A57.
Wastewater 5 - Third Fraction

(5μl, Col. 1, 200°C, 256X)

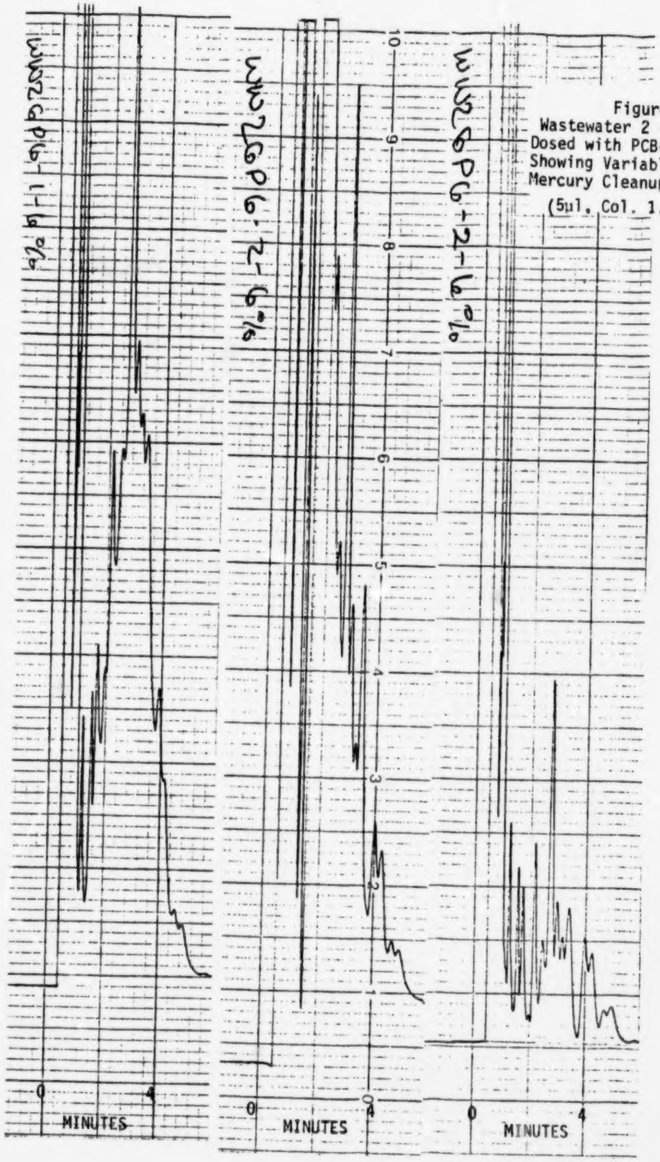


Figure A58.
Wastewater 2 - First Fractic
Dosed with PCB-1016
Showing Variable Response to
Mercury Cleanup.
(5µl, Col. 1, 200°C, 256X)

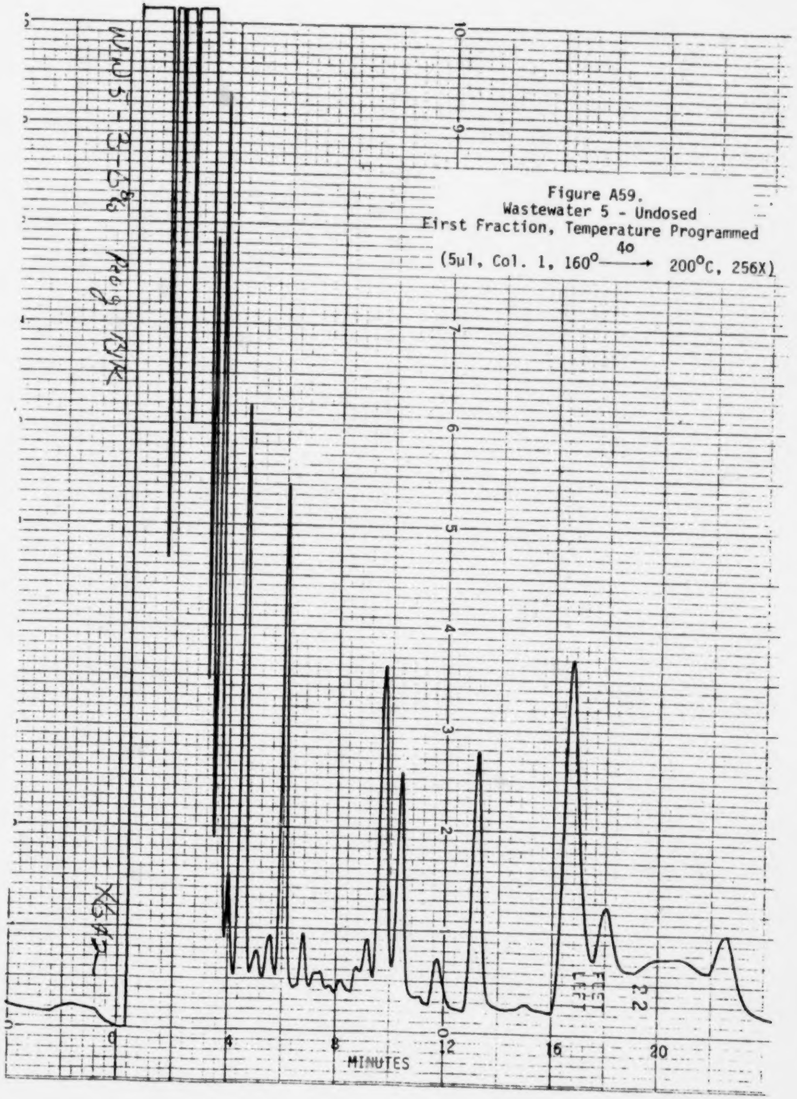


Figure A59.
Wastewater 5 - Undosed
First Fraction, Temperature Programmed
(5μl, Col. 1, 160° $\xrightarrow{4°}$ 200°C, 256X)

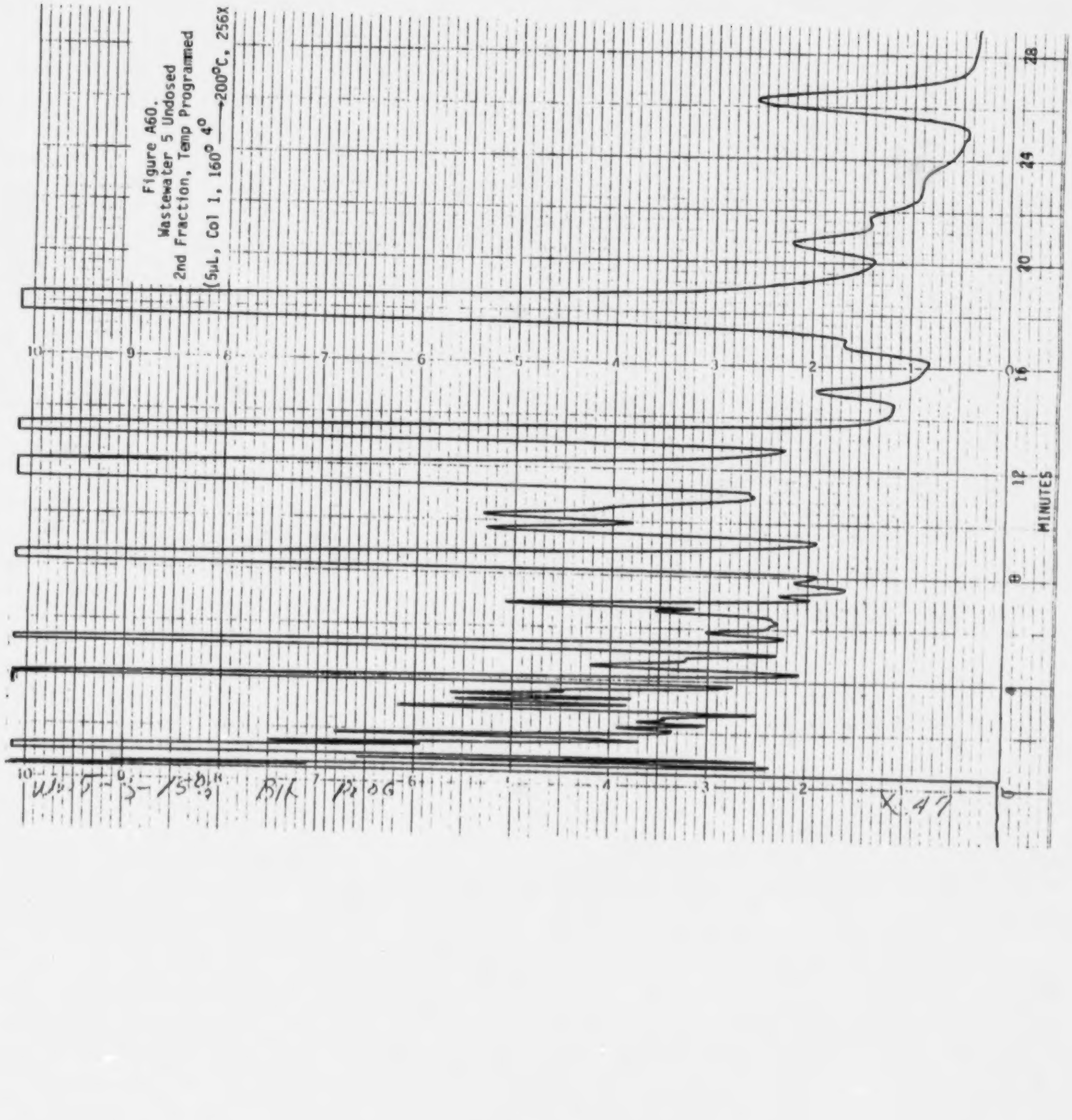


Figure A60.
Wastewater 5 Undosed
2nd Fraction, Temp Programmed
(5µL, Col 1, 160° 4° ⟶ 200°C, 256X

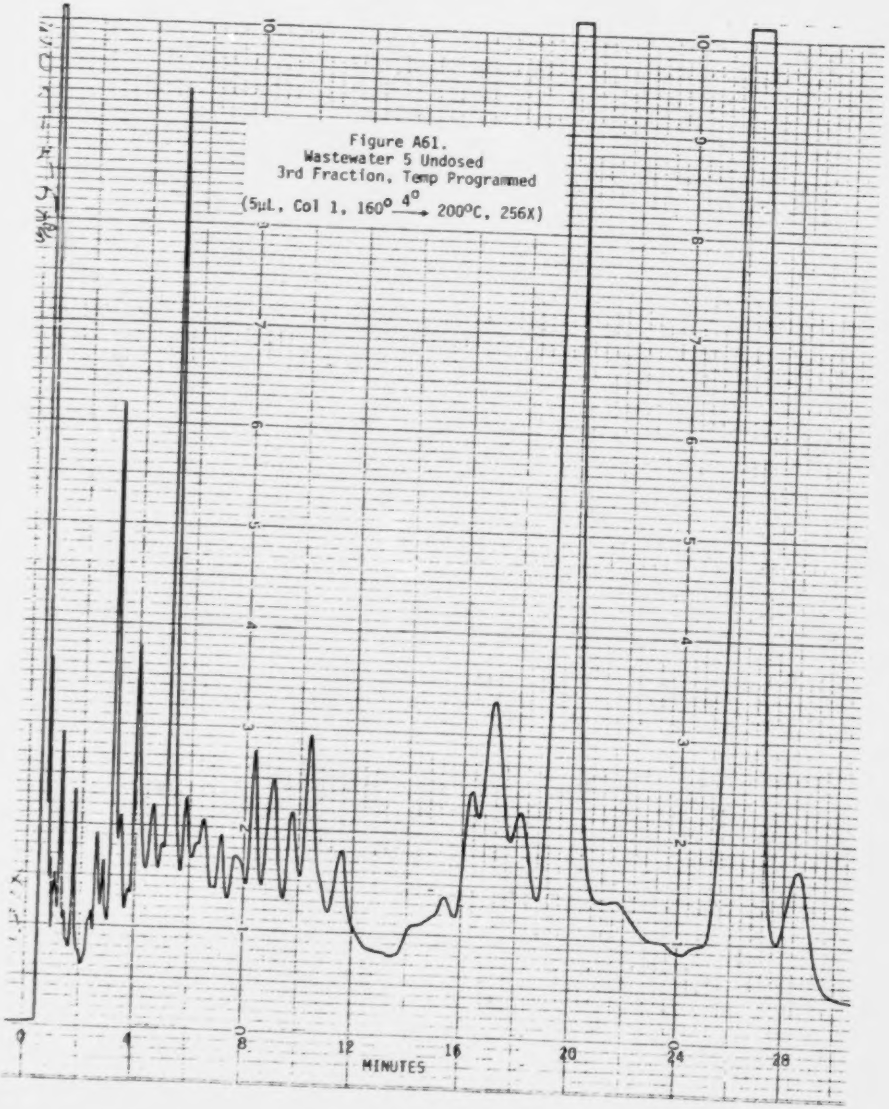


Figure A61.
Wastewater 5 Undosed
3rd Fraction, Temp Programmed
(5μL, Col 1, 160° $\xrightarrow{4^{\circ}}$ 200°C, 256X)

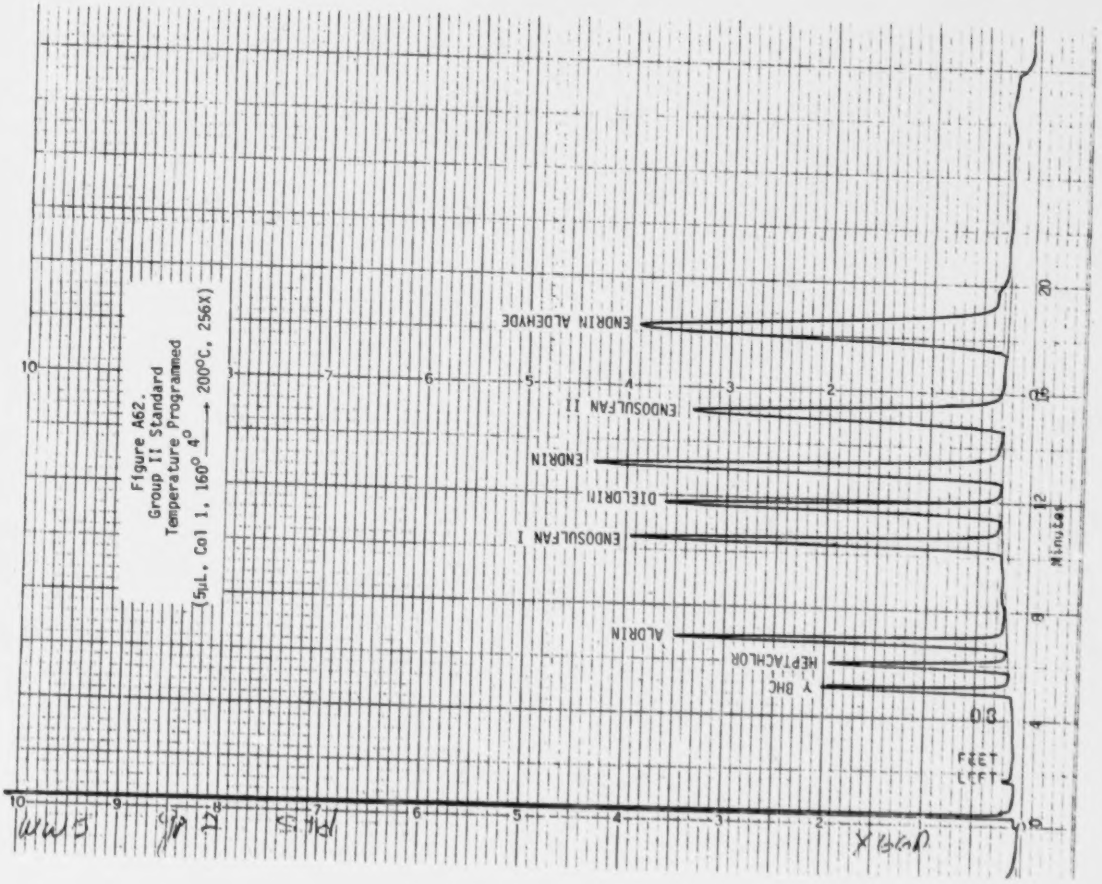


Figure A62.
Group II Standard
Temperature Programmed
(5µL, Col 1, 160° 4° → 200°C, 256X)

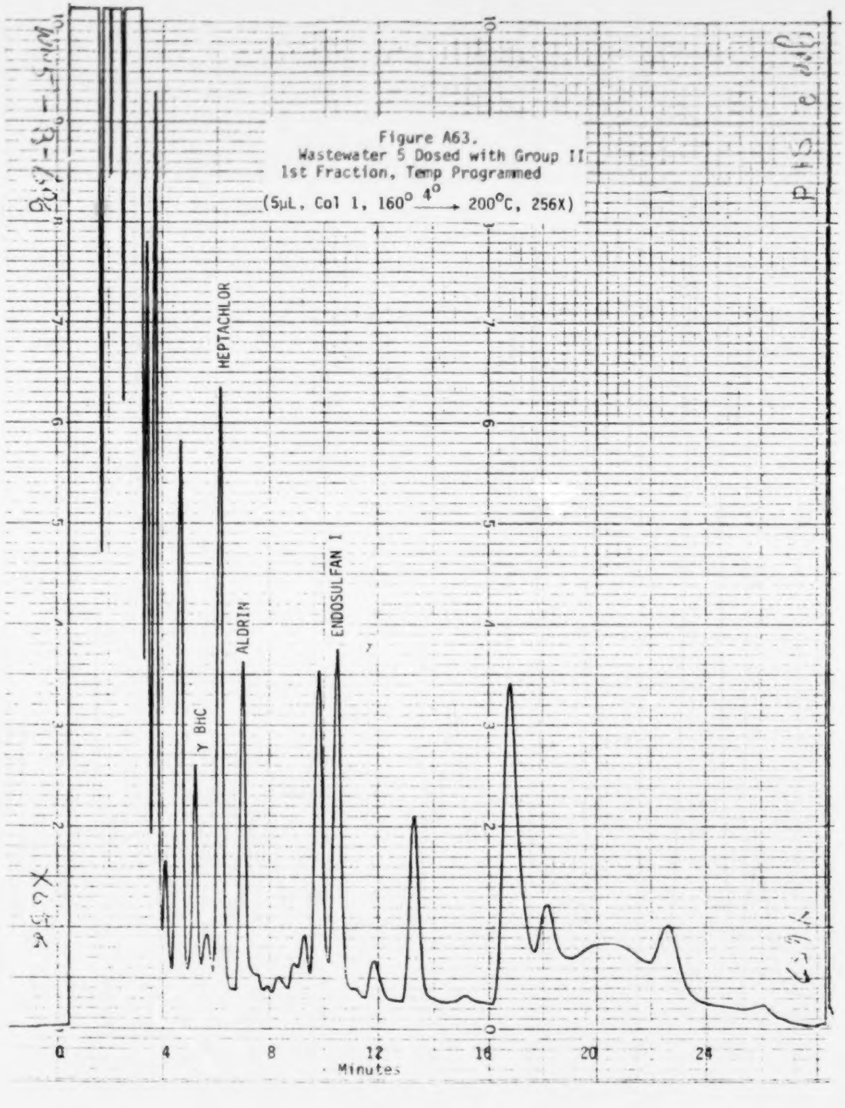


Figure A63.
Wastewater 5 Dosed with Group II
1st Fraction, Temp Programmed
(5μL, Col 1, 160° $\xrightarrow{4°}$ 200°C, 256X)

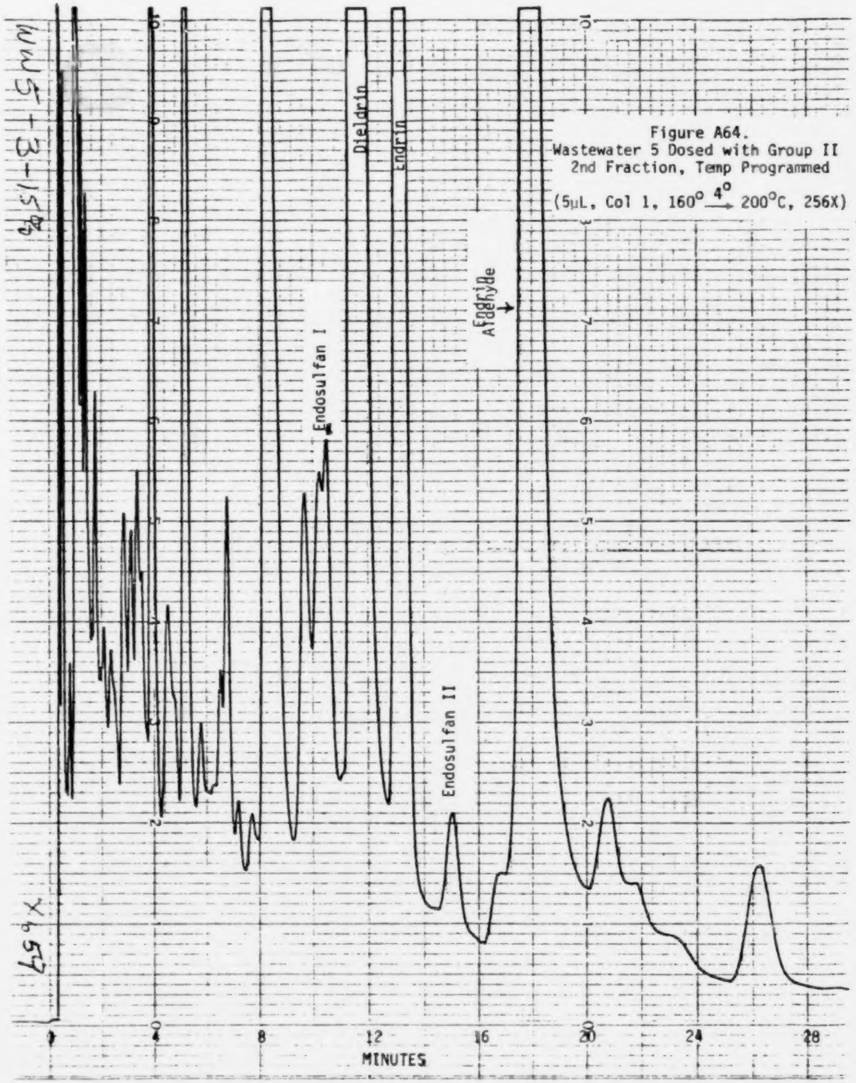


Figure A64.
Wastewater 5 Dosed with Group II
2nd Fraction, Temp Programmed
(5μL, Col 1, 160° $\xrightarrow{4^{\circ}}$ 200°C, 256X)

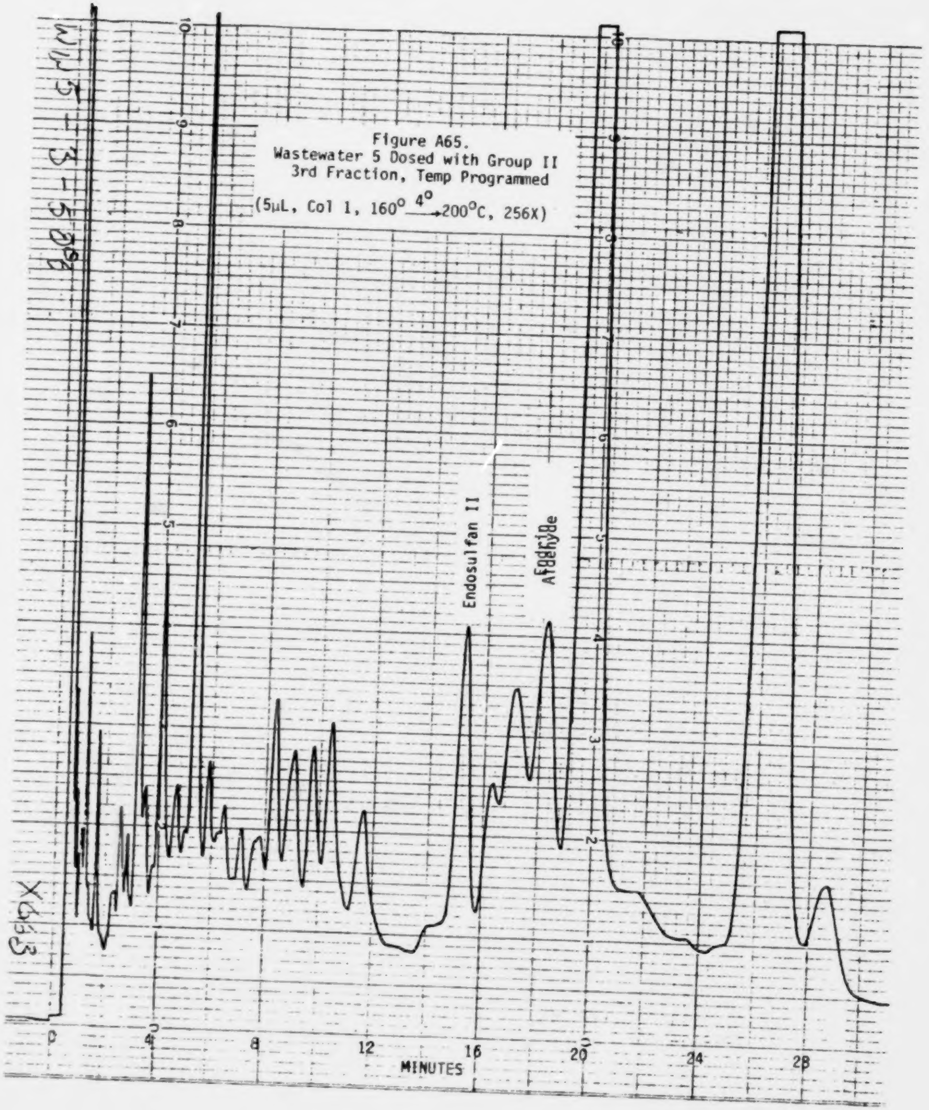


Figure A65.
Wastewater 5 Dosed with Group II
3rd Fraction, Temp Programmed
(5μL, Col 1, $160^{\circ} \xrightarrow{4^{\circ}} 200^{\circ}$C, 256X)

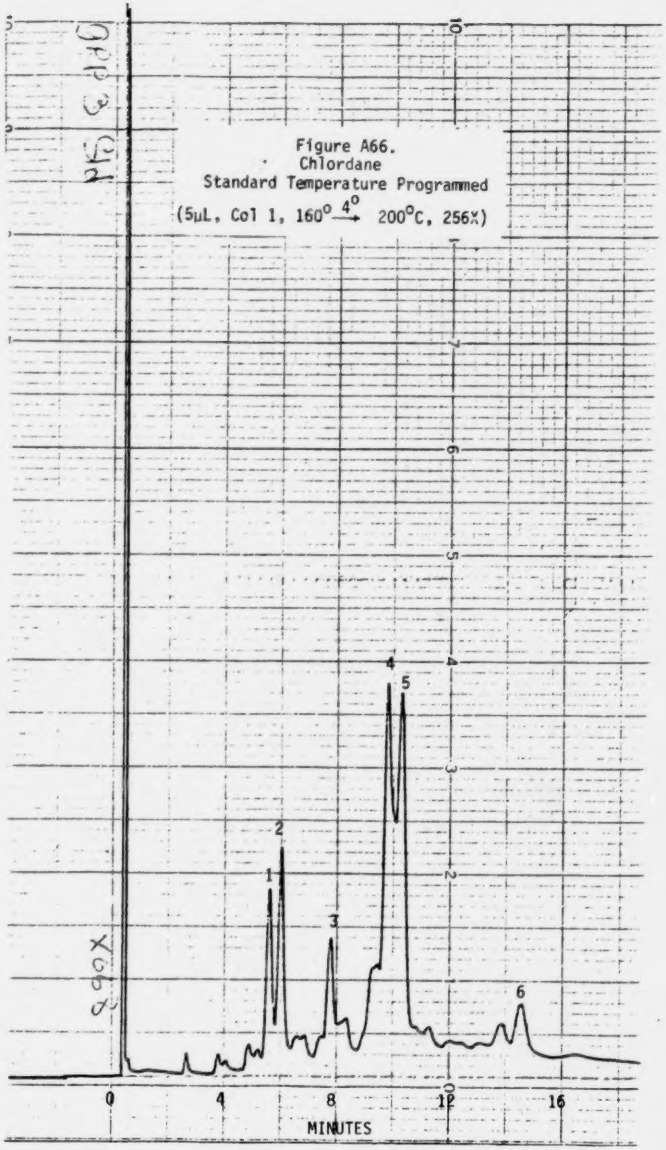


Figure A66.
Chlordane
Standard Temperature Programmed
(5μL, Col 1, 160° $\xrightarrow{4^\circ}$ 200°C, 256X)

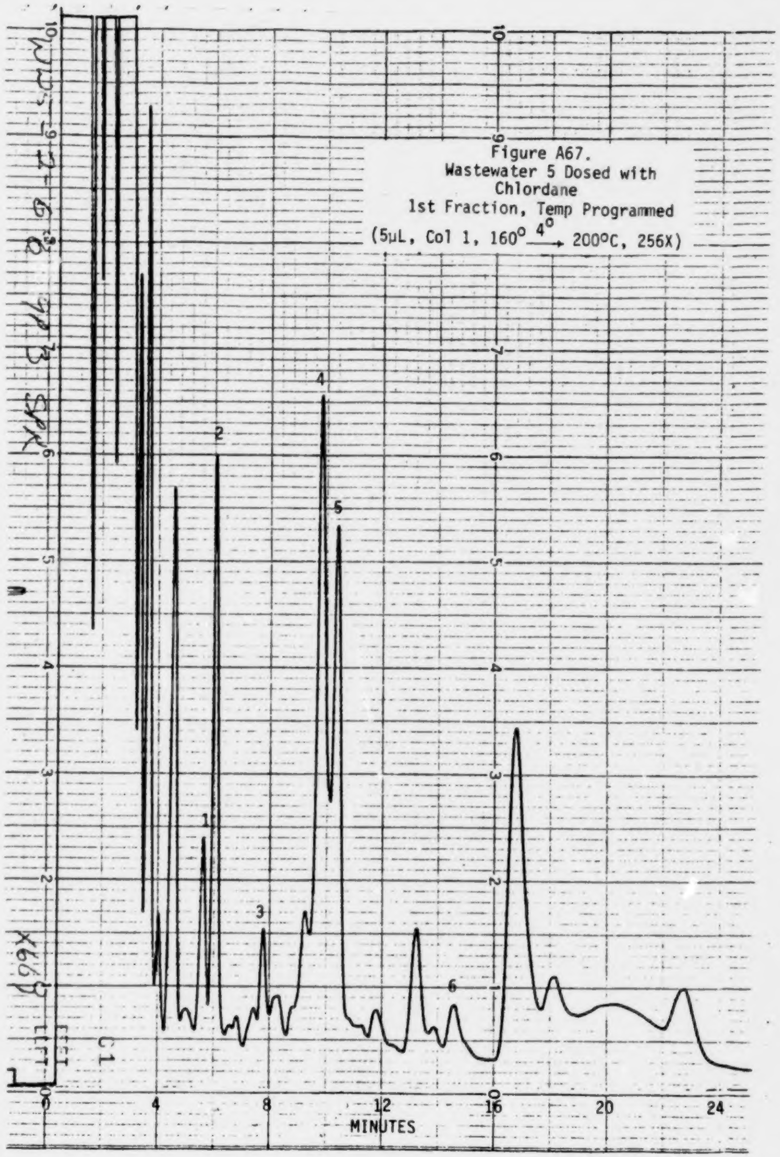


Figure A67.
Wastewater 5 Dosed with
Chlordane
1st Fraction, Temp Programmed
(5μL, Col 1, 160° $\xrightarrow{4^\circ}$ 200°C, 256X)

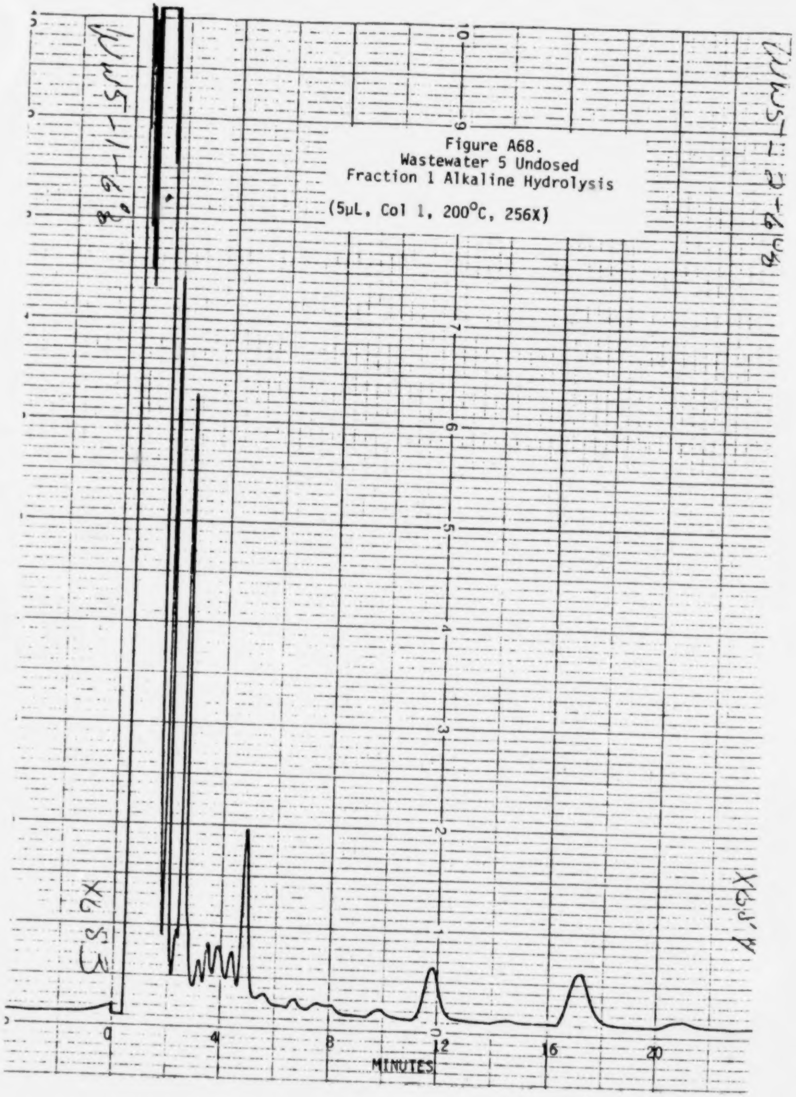


Figure A68.
Wastewater 5 Undosed
Fraction 1 Alkaline Hydrolysis

(5μL, Col 1, 200°C, 256X)

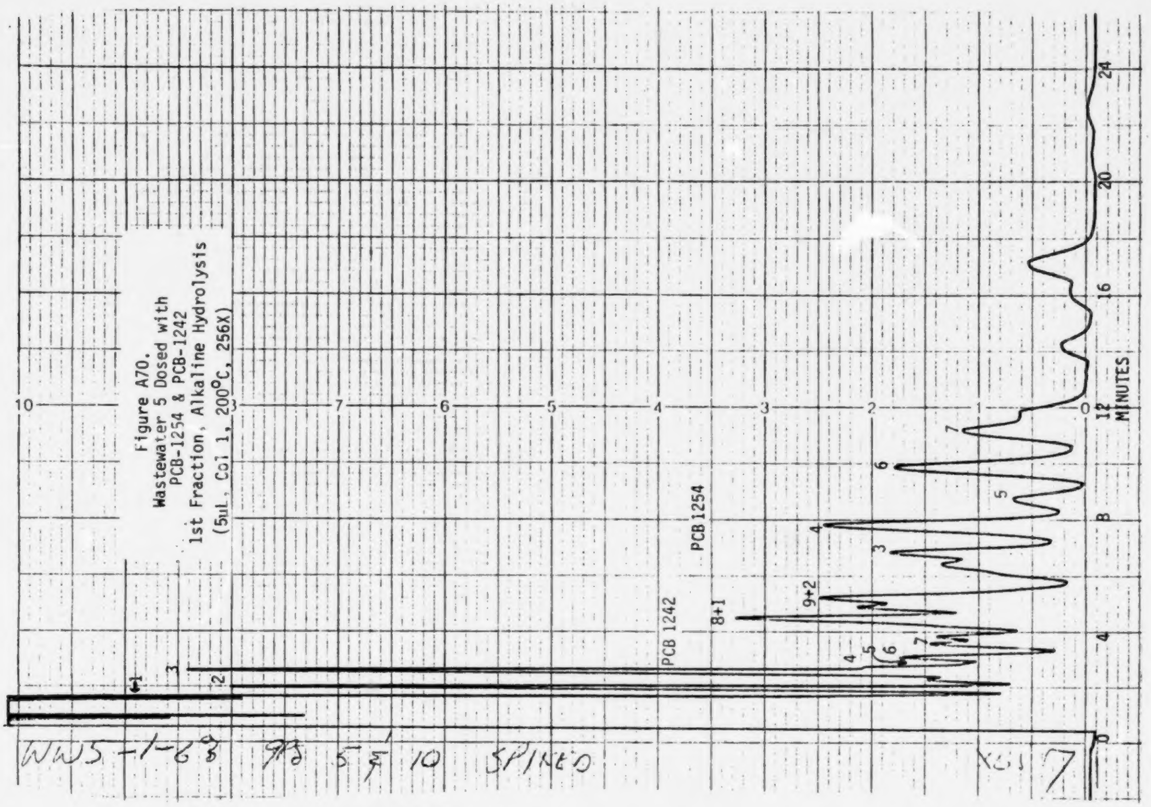


Figure A70.
Wastewater 5 Dosed with
PCB-1254 & PCB-1242
1st Fraction, Alkaline Hydrolysis
(5uL, Col 1, 200°C, 256X)

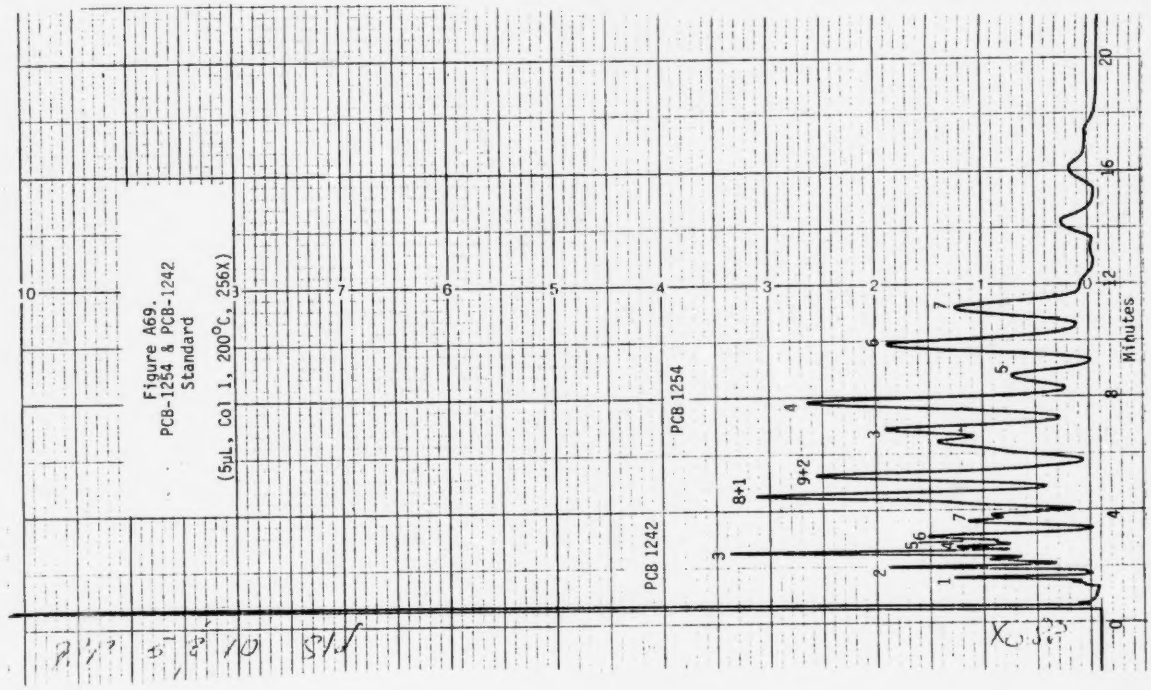


Figure A69.
PCB-1254 & PCB-1242
Standard
(5uL, Col 1, 200°C, 256X)

Table A60.
ANALYSIS OF WASTEWATER 1 FOR GROUP I, ppb

Replicate	alpha BHC	beta BHC	delta BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
1	.08	.03	.04	.07	.15	.02	0	0
2	.03	.05	.08	.11	.09	0	.02	0
3	.04	.04	.07	.07	.07	.03	0	0
"k",ppb, Avg.	.05	.04	.06	.08	.10	.02	.01	0

Note: No retention times coincide with standards.

Table A61.
ANALYSIS OF WASTEWATER 1 FOR GROUP II, ppb

Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Aldehyde
1	.04	.01	.03	.19	.16	.06	.06	.26
2	.03	.02	.07	.14	.10	.04	.02	.15
3	.03	.02	.06	.17	.10	0	.04	.16
"k",ppb, Avg.	.03	.02	.05	.17	.13	.03	.04	.19

note: Only retention times for heptachlor and endosulfan II are close enough to standards to be considered present on a tentative basis.

Table A62.
ANALYSIS OF WASTEWATER 1 FOR CHLORDANE, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
1	.31	.09	.35	0	.67	.40
2	.40	.32	1.64	.27	.77	0
3	.30	.20	0	0	.81	0
"k", ppb, Avg.	.34	.20	.66	.09	.75	.13

Note: Retention times do not coincide with standards

Table A63.
ANALYSIS OF WASTEWATER 1 FOR TOXAPHENE, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
1	11.65	0	1.03	1.35	0	4.73	0	0
2	2.72	3.15	0	1.42	0	0	.16	24.46
3	2.51	3.89	0	1.21	0	0	.80	1.16
"k", ppb, Avg.	5.63	2.35	.34	1.33	0	1.58	.32	8.54

Note: Only peak 4 coincides with standard

Table A64.
ANALYSIS OF WASTEWATER 1 FOR PCB-1254, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	.83	1.54	0	.52	0	.35	0
2	.82	.63	.79	.18	.39	.36	.24
3	.57	1.12	.97	.27	.48	.30	0
"k",ppb, Avg.	.74	1.10	.59	.32	.29	.34	.08

Note: Only peaks 1 and 2 coincide with standard

Table A65.
ANALYSIS OF WASTEWATER 1 FOR PCB-1016, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
1	4.88	1.24	.10	0	1.89	.46	0	1.73
2	4.70	1.07	.34	0	3.79	1.57	2.42	0
3	4.18	.99	.22	0	3.05	1.01	1.52	0
"k",ppb, Avg.	4.59	1.10	.33	0	2.91	1.01	1.31	.58

Note: Only peaks 3,6, and 7 coincide with standard.

Table A66.
ANALYSIS OF WASTEWATER 1 FOR PCB-1260, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10	Peak #11
1	14.84	10.41	.20	1.50	0	6.93	0	7.37	3.56	.13	0
2	11.56	13.85	1.87	.87	0	1.01	0	0	2.43	0	0
3	11.05	15.66	1.71	1.53	0	1.19	0	0	5.13	0	0
"k", ppb, Avg.	12.48	13.31	1.26	1.30	0	3.04	0	2.46	3.71	.04	0

Note: Retention times do not coincide with standard.

Table A67.
ANALYSIS OF WASTEWATER 1 FOR PCB-1221, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	7.63	7.18	5.08	11.83	4.10	5.26	.82
2	7.03	2.15	1.31	11.24	1.45	4.14	1.94
3	9.98	3.14	2.33	10.02	1.82	3.95	1.66
"k", ppb, Avg.	8.21	4.16	2.91	11.03	2.46	4.45	1.47

Note: Only peaks 2 and 5 coincide with standard.

Table A68.
ANALYSIS OF WASTEWATER 1 FOR PCB-1232, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10
1	297.65	62.09	14.55	41.35	8.41	0	5.43	4.25	8.22	15.14
2	200.53	45.59	6.13	9.01	6.40	0	9.28	8.36	8.47	17.96
3	187.26	50.59	8.59	10.32	6.92	5.12	0	6.40	7.24	20.23
"k", ppb Avg	228.48	52.76	9.76	20.23	7.24	1.71	4.90	6.34	7.98	17.78

Note: Only peaks 3 and 8 coincide with standard

Table A69.
ANALYSIS OF WASTEWATER 1 FOR PCB-1242, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9
1	5.18	1.33	.12	0	2.08	.42	1.16	1.14	.40
2	5.17	1.14	.27	0	3.41	1.02	.25	2.42	4.46
3	4.60	1.10	.26	2.48	0	1.28	1.92	1.68	4.52
"k", ppb, Avg.	4.98	1.19	.22	.83	1.83	.91	1.11	1.75	3.13

Note: Peaks 2, 3, 6, 7, 8 and 9 coincide with standard.

Table A70.
ANALYSIS OF WASTEWATER 1 FOR PCB-1248, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	3.47	.27	.74	0	1.11	.75	2.03
2	2.63	.59	.49	1.40	0	.74	.83
3	3.12	.56	.50	0	1.30	.52	1.47
"k", ppb, Avg.	3.07	.47	.58	.47	.80	.67	1.44

Note: Only peak 3 coincides with standard.

Table A71.
ANALYSIS OF WASTEWATER 2 FOR GROUP I, ppb

Replicate	alpha BHC	beta BHC	delta BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
1	0.420	6.991	0	0	0.014	0	0	2.254
2	0.459	14.588	0	0	0.007	0	0	0
3	0.489	15.549	0	0	0.012	0	0	0
"k" ppb, Avg.	.456	12.376	0	0	0.011	0	0	0.751

Note: Retention times do not coincide with standard.
No mercury clean up.

Table A72.
ANALYSIS OF WASTEWATER 2 FOR GROUP II, ppb

Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
1	1.510	3.021	0.613	0	0.873	0.028	0	0
2	0	6.135	0	0	0.838	0.020	0	0
3	0	6.812	0	0	0.885	0.013	0	0
"k",ppb, Avg.	0.503	5.322	0.204	0	0.865	0.020	0	0

Note: Retention times do not coincide with standard.
No mercury clean up.

Table A73.
ANALYSIS OF WASTEWATER 2 FOR CHLORDANE, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
1	33.055	43.053	0	0	0	0
2	0	84.518	0	0	0	0
3	0	0	0	0	0	0
"k", ppb, Avg.	11.018	75.339	0	0	0	0

Note: Retention times do not coincide with standard.
No mercury clean up.

Table A74.
ANALYSIS OF WASTEWATER 2 FOR TOXAPHENE, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #A+7	Peak #8
1	0	0	0	0	0	0	0.099	3.293
2	0	0	0	0	0	0	0.204	0
3	0	0	0	0	0	0	0.194	0
"k", ppb, Avg.	0	0	0	0	0	0	0.165	1.097

Note: Retention times do not coincide with standard.
No mercury clean up.

Table A75.
ANALYSIS OF WASTEWATER 2 FOR PCB-1254, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	0	0	0.231	0	0	0	0
2	0	0	0	0	0	0	0
3	0	0	0	0	0	0	0
"k", ppb, Avg.	0	0	0.077	0	0	0	0

Note: No mercury clean up.

Table A76.
ANALYSIS OF WASTEWATER 2 FOR PCB-1016, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
1	42.380	50.455	47.211	0	0	16.374	0	2.427
2	33.213	14.458	90.057	0	0	0	0	2.195
3	31.305	16.613	104.87	0	0	0	0	2.391
"k", ppb, Avg.	35.632	27.175	80.712	0	0	5.458	0	2.337

Note: Only peak 3 coincides with standard.
No mercury clean up.

Table A77.
ANALYSIS OF WASTEWATER 2 FOR PCB-1260, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10	Peak #11
1	0	4.956	28.297	0.242	0.616	0	0.678	1.024	17.311	0	0
2	0	0	28.958	0.266	0.353	0	0.613	0.858	10.334	0	0
3	0	4.189	33.207	0.203	0.697	0	0.783	0.937	14.488	0	0
"k", ppb, Avg.	0	3.048	30.154	0.237	0.622	0	0.691	0.939	14.044	0	0

Note: Retention times do not coincide with standard.
No mercury clean up.

Table A78.
ANALYSIS OF WASTEWATER 2 FOR PCB-1221, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	11,923	967	0	103	16	61	375
2	9,962	612	0	83	12	61	735
3	10,518	599	0	80	14	68	840
"k", ppb, Avg.	10,800	726	0	89	14	63	650

Note: Only peaks 4 and 7 coincide with standard.
No mercury clean up.

Table A79.
ANALYSIS OF WASTEWATER 2 FOR PCB-1232, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10
1	130,828	153	39	236	178	0	23	116	4.4	5
2	81,920	763	37	144	264	95	19	73	0	0
3	69,817	749	39	148	339	0	20	67	0	3.4
"k", ppb, Avg.	94,187	555	38	176	260	32	20	85	1.5	2.7

Note: Only peaks 2 and 5 coincide with standard.
No mercury clean up.

Table A80.
ANALYSIS OF WASTEWATER 2 FOR PCB-1242, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9
1	46	55	55	0	0	20	0	0	0
2	36	16	107	0	0	0	0	0	0
3	32	17	118	0	0	0	0	0	0
"k", ppb, Avg.	38	29	93	0	0	7	0	0	0

Note: Only peak 3 coincides with standard.
No mercury clean up.

Table A81.
ANALYSIS OF WASTEWATER 2 FOR PCB-1248, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	9	37	0	0	1	0	0
2	4	136	0	0	1.3	0	0
3	5	148	0	0	0	0	0
"k", ppb, Avg.	6	107	0	0	.7	0	0

Note: Only peak 2 coincides with standard.
No mercury clean up.

Table A82.
ANALYSIS OF WASTEWATER 3 FOR GROUP I, ppb

Replicate	alpha BHC	beta BHC	delta BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
1	.192	.172	.160	.028	.024	0	0	0
2	.128	.192	.132	.028	.024	0	0	.356
3	.196	.276	.112	.060	.020	0	0	.188
"k", ppb, Avg.	.154	.213	.134	.038	.022	0	0	.181

Note: Only peak for DDE coincides with standard.

Table A83.
ANALYSIS OF WASTEWATER 3 FOR GROUP II, ppb

Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
1	.040	.036	.084	.144	.052	0	1.060	0
2	.048	.020	.072	.016	.023	0	.483	0
3	.068	.028	.060	.160	.036	0	1.000	0
"k", ppb, Avg.	.052	.028	.072	.106	.038	0	.847	0

Note: Only the peak for dieldrin coincides with standards.

Table A84.
ANALYSIS OF WASTEWATER 3 FOR CHLORDANE, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
1	1.652	.296	.756	.652	.804	0
2	1.852	.248	.824	.200	.084	0
3	2.640	.372	.516	.836	.892	0
"k", ppb, Avg.	2.048	.305	.698	.562	.593	0

Note: Only peaks 4 and 5 coincide with standard.

Table A85.
ANALYSIS OF WASTEWATER 3 FOR TOXAPHENE, ppb

Replicate Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
1	.504	0	0	0	0	0	0	0
2	.352	0	0	0	0	0	.552	0
3	.344	0	0	0	0	0	.292	0
"k", ppb, Avg.	.400	0	0	0	0	0	.281	0

Note: Peaks do not coincide with standard.

Table A86.
ANALYSIS OF WASTEWATER 3 FOR PCB-1254, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	.540	.496	.808	.548	0	0	0
2	.516	.288	.564	.596	0	0	0
3	.488	.540	.552	0	0	0	0
"k", ppb, Avg.	.514	.441	.641	.381	0	0	0

Note: Only peaks 1 and 3 coincide with standard.

Table A87.
ANALYSIS OF WASTEWATER 3 FOR PCB-1016, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
1	36.524	2.952	.360	0	5.536	1.176	0	10.600
2	24.828	3.312	.300	0	4.648	1.280	0	8.296
3	44.100	3.724	.452	0	3.840	.804	.664	9.516
"k", ppb, Avg.	35.150	3.662	.370	0	4.674	1.120	.221	9.470

Note: Only peaks 3, 5 and 8 agree with standard.

ANALYSIS OF WASTEWATER 3 FOR PCB-1260, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10	Peak #11
1	21.948	27.756	.680	5.260	0	10.076	0	0	3.940	0	0
2	19.576	16.176	.496	3.664	0	.525	0	0	2.119	0	0
3	23.696	28.220	1.508	3.288	0	7.368	0	0	2.996	0	0
"k", ppb, Avg.	21.740	24.050	.894	4.070	0	5.989	0	0	3.018	0	0

Note: Retention times do not coincide with standard.

Table A89.
ANALYSIS OF WASTEWATER 3 FOR PCB-1221, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	62.832	3.316	0	95.732	12.632	13.252	3.164
2	5.680	5.028	0	65.076	8.464	14.852	2.652
3	12.844	0	0	115.592	13.012	21.192	3.968
"k", ppb, Avg.	27.118	2.781	0	92.133	11.369	16.432	3.261

Note: Only peaks 4 and 7 coincide with standard.

Table A90.
ANALYSIS OF WASTEWATER 3 FOR PCB-1232, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10
1	305.208	167.100	17.440	7.484	6.212	0	47.972	12.008	10.888	35.628
2	314.160	99.852	12.188	10.156	6.912	0	27.528	7.995	9.028	15.560
3	272.948	184.356	17.140	14.996	3.920	3.680	44.024	10.288	13.412	37.352
"k", ppb, Avg.	297.438	150.436	15.589	10.878	5.682	1.286	40.270	10.130	11.108	29.513

Note: Only peaks 2, 5, 7, 9, and 10 coincide with standard.

Table A91.
ANALYSIS OF WASTEWATER 3 FOR PCB-1242, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9
1	44.932	5.136	.480	0	7.608	1.632	12.756	0	1.880
2	30.496	4.104	.404	0	6.392	1.780	9.988	0	1.832
3	54.280	5.856	.256	0	4.504	1.036	11.056	0	1.540
"k", ppb Avg.	43.236	5.032	.380	0	6.168	1.482	11.266	0	1.750

Note: Only peaks 3 and 5 coincide with standard.

Table A92.
ANALYSIS OF WASTEWATER 3 FOR PCB-1248, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	9.460	1.072	2,480	0	10.288	.432	.712
2	17.276	.896	1,988	0	9,164	.416	.412
3	26,564	.948	1,520	.488	10.160	.392	.776
"k", ppb, Avg.	17.766	.972	1.996	.162	9,870	.413	.633

Note: Only peaks 2 and 6 coincide with standard.

Table A93.
ANALYSIS OF WASTEWATER 4 FOR GROUP I, ppb

Replicate	alpha BHC	beta BHC	delta BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
1	.396	.380	.536	.232	.088	.052	.116	1.760
2	.492	.592	.120	.184	.076	0	.056	1.888
3	.244	.640	.184	.216	.092	0	.100	5.676
4	.212	.648	.184	.216	.092	0	.100	4.496
5	.212	.257	.050	.086	.029	.037	0	1.735
6	.210	.244	.051	.089	.032	.032	.047	2.108
"k", ppb, Avg.	.211	.253	.051	.088	.031	.035	.024	1.922

Note: Only the peak for DDE coincides with standard.
Average for "k" is based on the last two replicates only (see text).

Table A94.
ANALYSIS OF WASTEWATER 4 FOR GROUP II, ppb

Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
1	.076	.340	.160	.708	.164	.068	1.552	0
2	.072	.276	.064	.572	.124	0	1.364	0
3	.054	.324	.120	.652	.160	.048	1.480	0
4	.044	.328	.120	.700	.384	.048	1.756	.516
5	.188	.121	.034	.270	.032	0	.622	0
6	.114	.117	.036	.292	.032	0	.618	0
"k", ppb, Avg.	.166	.119	.035	.281	.032	0	.620	0

Note: Only the peaks for heptachlor and dieldrin coincide with standard.
Average for "k" is based on the last two replicates only (see text).

Table A95.
ANALYSIS OF WASTEWATER 4 FOR CHLORDANE, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
1	3.916	4.620	5.700	3.356	4.008	1.220
2	4.656	3.656	4.108	3.292	3.232	0
3	3.592	4.272	3.912	3.776	3.688	0
4	3.376	4.336	3.760	3.960	3.940	.684
5	1.517	1.589	1.258	1.110	1.294	.844
6	1.551	1.544	1.192	1.173	1.370	.754
"k", ppb, Avg.	1.534	1.567	1.225	1.142	1.332	.799

Note: Peaks 1, 2, 3, 4 and 5 coincide with standard.
Average for "k" is based on the last two replicates only (see text).

Table A96.
ANALYSIS OF WASTEWATER 4 FOR TOXAPHENE, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
1	.848	4.474	1.736	0	0	0	.752	0
2	.460	1.984	0	0	0	0	0	0
3	.664	2.024	0	0	0	0	.632	0
4	.712	2.720	.904	0	1.324	0	1.140	0
5	.247	.796	1.143	0	0	0	.738	0
6	.251	.800	1.021	0	0	0	3.664	0
"k", ppb, Avg.	.249	.798	1.082	0	0	0	1.832	0

Note: Retention times do not coincide with standard.
Average for "k" is based on the last two replicates only (see text).

Table A97.
ANALYSIS OF WASTEWATER 4 FOR PCB-1254, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	1.556	5.452	1.356	.800	4.428	0	1.840
2	1.268	1.768	.736	.436	2.876	0	.724
3	4.640	8.720	4.256	2.176	11.760	0	6.176
4	1.140	2.228	1.144	.568	3.328	0	.636
5	.541	1.720	.404	.183	0	0	0
6	.532	1.775	.410	.188	0	0	0
"k", ppb, Avg.	.537	1.748	.407	.186	0	0	0

Note: Only peaks 1 and 4 coincide with standard.
Average for "k" based on the last two replicates only (see text).

Table A98.
ANALYSIS OF WASTEWATER 4 FOR PCB-1016, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
1	24.200	18.936	5.776	0	10.720	3.492	12.300	27.956
2	24.156	20.812	4.440	0	4.172	1.512	8.516	23.680
3	20.816	3.836	5.188	0	7.656	2.424	8.108	19.300
4	18.116	3.008	5.268	0	7.624	2.864	7.788	23.856
5	17.327	6.416	2.118	0	1.862	.975	2.628	5.468
6	17.405	3.885	2.058	0	1.884	1.057	2.490	5.667
"k", ppb, Avg.	17.366	5.151	2.088	0	1.873	1.016	2.559	5.568

Note: Only peaks 5 and 8 coincide with standard.
Average for "k" is based on the last two replicates only (see text).

Table A99.
ANALYSIS OF WASTEWATER 4 FOR PCB-1260, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10	Peak #11
1	67.916	79.916	1.196	8.796	1.736	2.024	2.260	0	28.488	0	0
2	56.244	67.624	.648	6.584	1.128	6.344	.888	0	20.224	0	0
3	85.516	25.808	.936	8.072	1.152	3.492	1.896	0	8.524	0	0
4	148.572	26.408	1.008	16.720	1.304	15.464	.780	0	24.572	0	0
5	3.723	7.870	.326	.464	.353	0	0	0	.522	.371	0
6	3.665	8.119	.330	.476	.383	0	.837	0	.449	.254	0
"k", ppb. Avg.	3.694	7.995	.328	.470	.368	0	.419	0	.486	.313	0

Note: Retention times dp not coincide with standard.
Average for "k" is based on the last two replicates only (see text).

Table A100.
ANALYSIS OF WASTEWATER 4 FOR PCB-1221, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	14.076	14.240	0	63.428	0	84.972	50.764
2	79.536	7.068	0	65.936	0	93.388	39.024
3	17.300	11.896	5.104	54.608	10.200	17.208	45.592
4	14.832	7.988	4.004	47.484	10.328	13.580	46.312
5	7.994	0	0	38.967	0	24.313	15.614
6	7.340	0	0	39.142	2.871	14.721	15.171
"k", ppb, Avg.	7.667	0	0	39.055	1.436	19.517	15.393

Note: Only peak 4 coincides with standard.
Average for "k" is based on the last two replicates only (see text).

Table A101.
ANALYSIS OF WASTEWATER 4 FOR PCB-1232, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10
1	397.344	99.020	1.700	50.312	23.720	20.600	64.024	42.996	53.716	144.528
2	522.784	101.168	2.196	55.804	16.848	15.336	37.572	25.936	40.672	100.852
3	361.616	91.244	12.760	10.188	20.224	21.352	42.068	35.748	52.428	136.740
4	585.812	81.524	12.920	9.352	14.508	44.676	47.644	67.768	77.312	137.808
5	17.612	58.877	0	14.513	5.822	0	5.056	3.020	4.067	18.702
6	18.713	59.141	3.689	8.788	5.649	0	5.083	3.251	4.222	19.402
"k" ppb, Avg.	18.163	59.009	1.845	11.651	5.736	0	5.070	3.136	4.145	19.052

Note: Only peaks 2, 7, and 9 coincide with the standard.
Average for "k" is based on the last two replicates only (see text).

Table A102.
ANALYSIS OF WASTEWATER 4 FOR PCB-1242, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9
1	29.784	23.484	7.332	0	14.740	4.856	17.132	8.104	43.384
2	30.960	25.808	5.944	0	5.736	2.104	28.500	5.800	28.888
3	26.348	4.756	6.928	0	10.444	3.316	23.008	7.312	32.340
4	22.852	3.752	6.984	0	10.148	3.752	27.764	1.860	28.080
5	18.962	7.058	2.511	0	2.276	1.245	5.997	2.486	9.986
6	19.046	4.273	2.436	0	2.289	1.340	6.173	2.548	10.053
"k", ppb, Avg.	19.004	5.666	2.474	0	2.283	1.293	6.085	2.517	10.020

Note: Only peaks 5 and 8 coincide with the standard.
Average for "k" is based on the last two replicates only (see text).

Table A103.
ANALYSIS OF WASTEWATER 4 FOR PCB-1248, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	51.840	5.792	2.036	8.932	28.680	1.252	3.444
2	44.988	5.696	1.132	4.738	21.252	1.020	2.540
3	18.072	5.388	1.216	3.440	15.997	.932	3.136
4	18.508	5.592	1.564	3.304	20.744	.920	3.200
5	15.330	1.972	.494	1.070	4.129	.390	.775
6	8.716	1.971	.604	1.068	4.341	.384	.785
"k", ppb, Avg.	12.023	1.972	.549	1.069	4.235	.387	.780

Note: Only peaks 5 and 6 coincide with the standard.
Average for "k" is based on the last two replicates only (see text).

Table A104.
ANALYSIS OF WASTEWATER 5 FOR GROUP I, ppb

Replicate	alpha BHC	beta BHC	delta BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
1	2.891	14.722	.166	.446	0	0	0	5.250
2	3.202	13.702	0	.517	0	0	8.241	10.824
3	3.373	14.038	.047	.452	0	0	8.443	11.191
"k", ppb, Avg.	3.155	14.154	.071	.471	0	0	5.561	9.088

Note: Only the peak for heptachlor epoxide coincides with standard.

Table A105.
ANALYSIS OF WASTEWATER 5 FOR GROUP II, ppb

Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
1	1.736	6.887	.120	7.157	68.380	32.882	2.328	97.224
2	1.922	6.409	.304	7.053	65.974	31.665	2.499	93.290
3	2.026	6.567	.160	1.747	62.805	30.559	1.665	85.576
"k", ppb, Avg.	1.894	6.621	.194	5.319	65.719	31.702	2.164	92.030

Note: Only the peaks for heptachlor and endrin coincide with the retention times of standards.

Table A106.
ANALYSIS OF WASTEWATER 5 FOR CHLORDANE, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
1	4.927	90.671	2.314	12.325	6.518	0
2	5.217	84.387	5.885	14.336	9.553	0
3	4.798	86.456	3.092	14.498	9.860	0
"k", ppb, Avg.	4.980	87.171	3.763	13.719	8.643	0

Note: Only peak 2 coincides with the standard.

Table A107.
ANALYSIS OF WASTEWATER 5 FOR TOXAPHENE, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
1	11.493	0	0	3.038	0	18.029	18.352	0
2	9.452	0	0	3.891	0	0	19.979	0
3	10.877	0	0	3.155	0	0	19.858	0
"k", ppb, Avg.	10.607	0	0	3.361	0	6.009	19.396	0

Note: Only peaks 1 and 3 coincide with standard.

Table A108.
ANALYSIS OF WASTEWATER 5 FOR PCB-1254, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	4.066	10.013	18.426	17.078	0	2.632	0
2	3.580	18.230	14.956	2.126	0	2.687	77.380
3	3.363	19.628	17.438	7.075	0	2.719	80.699
"k", ppb, Avg.	3.669	15.957	16.940	8.759	0	2.679	52.693

Note: Retention times do not coincide with standard.

Table A109.
ANALYSIS OF WASTEWATER 5 FOR PCB-1016, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
1	4837.15	122.160	110.157	0	5.755	3.600	4.103	9.000
2	5105.466	135.269	102.522	0	0	9.157	6.001	12.101
3	4976.404	142.523	105.036	0	1.631	4.811	3.915	9.166
"k", ppb, Avg.	4973.006	133.317	105.905	0	2.462	5.856	4.673	10.089

Note: Retention times do not coincide with standard.

Table A110.
ANALYSIS OF WASTEWATER 5 FOR PCB-1260, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10	Peak #11
1	590.372	295.003	0	971.755	0	37.152	7.199	25.458	97.308	0	.053
2	581.008	320.767	0	946.103	0	37.983	99.895	0	53.843	2.039	2.394
3	564.080	310.185	0	900.452	0	36.542	102.716	0	43.134	3.607	.568
"k", ppb, Avg.	578.5	308.7	0	939.4	0	37.2	69.9	8.5	64.8	1.9	1.0

Note: Only peak 6 coincides with standard.

Table A111.
ANALYSIS OF WASTEWATER 5 FOR PCB-1221, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	26,166.7	7486.69	34,340.7	0	2.363	548.132	968.060
2	26,286.360	7719.275	36,351.510	0	0	606.954	900.959
3	26,348.705	7248.625	35,787.230	0	0	639.501	923.056
"k", ppb. Avg.	26,267.3	7484.9	35,493.1	0	.8	598.2	930.7

Note: Only peak 3 coincides with standard.

Table A112.
ANALYSIS OF WASTEWATER 5 FOR PCB-1232, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10
1	68,856.42	21,945.33	1629.931	7649.489	368.054	0	525.95	12.460	206.207	649.989
2	67,963.91	23,014.71	6147.962	1018.849	342.869	0	568.536	31.690	236.569	672.440
3	67,698.10	22,319.26	1578.960	7313.717	344.828	0	507.143	16.651	215.287	662.235
"k", ppb, Avg.	68,172.8	22,426.4	3119.0	5327.4	351.9		533.9	20.3	219.4	661.6

Note: Only peak 3 coincides with standard.

Table A113.
ANALYSIS OF WASTEWATER 5 FOR PCB-1242, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9
1	6342.68	151.480	147.470	0	7.912	5.005	5.715	14.080	58.268
2	6725.974	167.736	137.248	0	0	12.730	8.360	16,336	85.396
3	6489.359	176.730	140.614	0	2.243	6.689	5.454	14.258	88.144
"k", ppb, Avg.	6519.3	165.3	141.8	0	3.4	8.1	6.5	14.9	77.3

Note: Retention times do not coincide with standard.

Table A114.
ANALYSIS OF WASTEWATER 5 FOR PCB-1248, ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	391.312	315.000	5.540	2.205	3.433	3.274	14.387
2	463.644	285.559	7.780	2.100	3.704	2.883	26.195
3	456.540	299.291	6.954	2.102	3.342	2.708	28.203
"k", ppb, Avg.	437.2	300.2	6.8	2.1	3.5	3.0	22.9

Note: Retention times do not coincide with standard.

Table A115.
ANALYSIS OF WASTEWATER 1 DOSED WITH GROUP I.

	Dose, ppb							
	1	2	2	2	3	6	8	15
Replicate	alpha BHC	beta BHC	delta BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
Zero Time								
1								
Method Recovery, %	1.08 103	1.90 93	1.97 94	2.08 100	3.13 101	5.90 98	8.78 110	16.52 110
2								
Method Recovery, %	1.09 104	1.90 93	2.15 105	2.08 100	3.09 100	6.11 102	8.11 101	16.03 107
3								
Method Recovery, %	1.04 99	1.91 94	1.95 95	2.03 98	2.97 96	5.83 97	8.01 100	14.83 99
Xo Avg.	1.07	1.90	2.02	2.06	3.06	5.95	8.30	15.79
k	.05	.04	.06	.08	.10	.02	.01	0
Method Recovery, % Avg.	102	93	98	99	99	99	104	105
7 days at 4°C								
1								
Preservation Recovery, %	.99 93	1.84 97	1.85 92	1.98 96	2.97 97	5.73 96	8.[illegible] 98	14.31 91
2								
Preservation Recovery, %	.98 92	1.81 95	1.82 90	1.93 94	2.80 92	5.53 93	7.90 95	14.09 89
3								
Preservation Recovery, %	1.00 93	1.84 97	1.87 93	1.99 97	2.99 98	5.71 96	8.22 99	14.00 89
Preservation Recovery, % Avg.	93	96	92	96	96	95	97	90

Table A116.
ANALYSIS OF WASTEWATER 1 DOSED WITH GROUP II

	Dose, ppb							
	1	1	2	3	3	5	5	12
Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
Zero Time								
1 Method Recovery, %	.98 95	1.03 101	1.98 97	3.04 96	2.92 93	4.74 95	5.19 103	11.18 92
2 Method Recovery, %	.95 92	.95 93	1.90 93	3.17 100	2.79 89	4.75 95	*4.37 87	*9.76 80
3 Method Recovery, %	.89 86	.94 92	1.82 89	3.00 94	3.10 99	4.78 96	5.86 116	11.89 98
Xo Avg.	.94	.97	1.90	3.07	2.94	4.76	(5.53)	(11.54)
k	.03	.02	.05	.17	.11 .02	0	.04	.16
Method Recovery, % Avg.	91	95	93	97	97	95	(110)	(95)
7 days at 4°C								
1 Preservation Recovery, %	.91 97	.94 97	1.83 96	2.90 94	2.74 93	4.48 94	4.90 (89)	10.57 (92)
2 Preservation Recovery, %	.91 97	.93 96	1.80 95	2.94 96	2.78 95	4.66 98	5.00 (90)	11.04 (96)
3 Preservation Recovery, %	.97 103	.99 102	1.90 100	3.04 99	2.85 97	4.85 102	5.07 (92)	10.64 (92)
Preservation Recovery, % Avg.	99	98	97	96	95	98	(90)	(93)

Note: * lost small amount; leak around K-D joint on fraction 3; these values were excluded from all calculations in parentheses.

Table A117.
ANALYSIS OF WASTEWATER 1 DOSED WITH CHLORDANE

	Dose - 20 ppb					
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
Zero Time						
1	18.53	21.42	19.17	19.77	18.87	20.39
Method Recovery, %	91	106	93	98	91	101
2	17.40	18.08	21.26	18.52	18.51	22.82
Method Recovery, %	85	89	103	92	89	113
3	17.97	18.56	22.20	19.04	19.12	20.81
Method Recovery, %	88	92	108	95	92	103
Xo Avg.	17.97	19.35	20.88	19.11	18.83	21.34
k	0.34	0.20	0.66	0.09	0.75	0.13
Method Recovery, % Avg.	88	96	101	95	90	106
7 days at 4°C						
1	17.93	21.04	19.03	19.15	19.11	21.74
Preservation Recovery, %	100	109	91	100	101	102
2	18.52	16.72	19.65	18.13	19.14	24.26
Preservation Recovery, %	103	86	94	95	102	114
3	18.56	21.91	19.71	18.10	19.72	23.83
Preservation Recovery, %	103	113	94	95	105	112
Preservation Recovery, % Avg.	102	103	93	97	103	109

Table A118.

ANALYSIS OF WASTEWATER 1 DOSED WITH TOXAPHENE

	Dose - 200 ppb							
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
Zero Time								
1	199.70	208.52	209.92	200.52	200.56	201.83	202.04	207.56
Method Recovery, %	97	103	105	100	100	100	101	100
2	204.68	209.69	215.57	199.97	214.20	205.17	207.38	208.31
Method Recovery, %	100	104	108	99	107	102	104	100
3	206.32	210.74	206.22	202.97	213.44	207.00	204.06	205.90
Method Recovery, %	100	104	103	101	107	103	102	99
Xo Avg.	203.57	209.65	210.57	201.15	209.43	204.67	204.61	207.26
k	5.63	2.35	0.34	1.33	0	1.58	0.32	8.54
Method Recovery, % Avg.	99	104	105	100	105	102	102	104
7 days at 4°C								
1	203.07	206.91	206.71	203.14	217.47	208.32	212.19	211.47
Preservation Recovery, %	100	99	98	101	104	102	104	102
2	206.52	208.54	207.60	204.39	225.68	210.46	215.35	214.81
Preservation Recovery, %	101	99	99	102	108	103	105	104
3	203.38	202.80	194.16	199.19	220.24	207.30	207.18	206.29
Preservation Recovery, %	99	97	92	99	105	101	101	100
Preservation Recovery, % Avg.	100	98	96	101	106	102	103	102

Table A119.

ANALYSIS OF WASTEWATER 1 DOSED WITH PCB-1254

Replicate	Dose - 40 ppb						
	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
Zero Time							
1	37.68	38.58	37.87	36.90	36.59	36.67	36.78
Method Recovery, %	92	94	93	91	91	91	92
2	39.02	38.88	38.89	36.85	39.01	37.56	41.88
Method Recovery, %	96	94	96	91	97	93	105
3	39.96	41.02	39.88	39.87	40.64	38.70	39.34
Method Recovery, %	98	100	98	99	101	96	98
Xo Avg.	38.89	39.49	38.29	37.87	38.75	37.64	39.33
k	.74	1.10	.59	.32	.29	.34	0.08
Method Recovery, % Avg.	95	96	96	94	96	93	98
7 days at 4°C							
1*	38.42	39.01	38.99	37.95	36.62	37.03	37.96 **
Preservation Recovery, %	99	99	102	100	95	98	97
2	41.16	41.91	41.20	40.85	40.86	39.74	41.23
Preservation Recovery, %	106	106	108	108	105	106	105
3	40.66	41.31	40.82	40.42	40.52	39.46	41.90
Preservation Recovery, %	105	105	107	107	105	105	107
Preservation Recovery, % Avg.	103	103	106	105	102	103	103

Note; *Sample volume estimated because of spill.
**From peak height measurement.

Table A120.
ANALYSIS OF WASTEWATER 1 DOSED WITH PCB-1016

Replicate	Dose - 25 ppb							
	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
Zero Time								
1	26.08	24.17	24.69	24.00	24.45	24.03	24.54	23.92
Method Recovery, %	86	92	97	96	86	92	93	93
2	25.64	23.57	24.03	23.35	23.81	23.49	23.53	23.15
Method Recovery, %	84	90	95	93	84	90	89	90
3	27.12	24.67	25.17	24.47	24.97	24.54	24.71	24.47
Method Recovery, %	90	94	99	98	88	94	94	96
Xo Avg.	26.28	24.14	24.63	23.94	24.41	24.02	26.26	23.85
k	4.59	1.10	0.33	0	2.91	1.01	1.31	0.58
Method Recovery, % Avg.	87	92	97	96	86	92	92	93
7 days at 4°C								
1	29.03	25.53	24.40	24.05	23.95	24.20	23.83	23.85
Preservation Recovery, %	110	106	99	100	98	101	98	100
2	26.55	24.10	24.07	23.53	23.68	23.42	23.48	23.48
Preservation Recovery, %	101	100	98	98	97	98	97	98
3	27.74	25.01	24.91	24.38	24.64	24.47	24.38	24.66
Preservation Recovery, %	106	104	101	102	105	102	100	103
Preservation Recovery, % Avg.	106	103	99	100	100	100	98	100

Table A121.
ANALYSIS OF WASTEWATER 1 DOSED WITH PCB-1260

	Dose - 80 ppb										
Replicate	Peak#1	Peak#2	Peak#3	Peak#4	Peak#5	Peak#6	Peak#7	Peak#8	Peak#9	Peak#10	Peak#11
Zero Time											
1	93.20	94.23	79.86	80.07	78.82	78.91	80.21	75.04	76.62	74.00	73.84
Method Recovery, %	101	102	98	98	99	95	100	91	93	92	92
2	99.49	95.73	77.19	77.00	75.27	75.09	75.87	72.32	73.30	71.37	71.68
Method Recovery, %	109	103	95	95	94	90	95	87	87	89	90
3	89.03	89.68	77.61	77.68	77.62	77.45	79.51	75.92	78.74	76.23	75.76
Method Recovery, %	96	95	95	95	97	93	99	92	94	95	95
Xo Avg.	93.91	93.21	78.22	78.25	77.24	77.15	78.53	74.43	76.22	73.87	73.76
k	12.48	13.31	1.26	1.30	0	3.04	0	2.46	3.71	0.04	0
Method Recovery, % Avg.	102	100	96	96	97	93	98	90	91	92	92
7 days at 4°C											
1	89.87	93.10	81.70	81.97	83.14	83.21	84.32	83.84	84.86	83.09	83.06
Preservation Recovery, %	96	100	104	105	108	108	107	113	111	112	113
2	93.56	95.49	83.22	83.44	84.94	85.26	86.54	85.52	86.32	85.06	84.08
Preservation Recovery, %	100	102	106	107	110	111	100	115	113	115	114
3	89.41	89.56	80.31	79.98	80.70	81.34	81.96	81.28	82.72	80.39	79.56
Preservation Recovery, %	95	96	103	102	104	105	104	109	109	109	108
Preservation Recovery, % Avg.	97	99	105	105	107	108	107	112	111	112	111

Note: The fifteen percent Et_2O fraction appears to have no Group VII unit. All values are from the 6% Et_2O fraction only.

Table A122.
ANALYSIS OF WASTEWATER 1 DOSED WITH PCB-1221

	Dose - 55 ppb						
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
Zero Time							
1	58.85	67.10	62.02	54.74	56.71	58.08	53.35
Method Recovery, %	92	114	107	98	99	98	94
2	59.51	68.20	63.14	64.41	56.54	58.08	53.35
Method Recovery, %	93	116	110	97	98	98	94
3	62.26	70.68	66.39	65.89	57.64	58.80	53.96
Method Recovery, %	98	121	115	100	100	99	95
Xo Avg.	60.21	68.66	63.85	65.01	56.96	58.32	53.55
k	8.21	4.16	2.91	11.03	2.46	4.45	1.47
Method Recovery, % Avg.	95	117	111	98	99	98	95
7 days at 4°C							
1	61.00	68.75	63.42	65.40	58.80	60.23	55.00
Preservation Recovery, %	101	100	99	100	103	103	103
2	59.68	73.32	67.65	66.11	58.80	61.55	56.10
Preservation Recovery, %	99	107	106	102	103	106	105
3	55.00	63.14	57.81	59.84	53.74	55.00	50.88
Preservation Recovery, %	91	92	89	92	94	94	95
Preservation Recovery, % Avg.	97	100	98	98	100	101	101

Note: all values are from peak height measurements.

Table A123.
ANALYSIS OF WASTEWATER 1 DOSED WITH PCB-1232

	Dose - 110ppb									
Replicate	Peak#1	Peak#2	Peak#3	Peak#4	Peak#5	Peak#6	Peak#7	Peak#8	Peak#9	Peak#10
Zero Time										
1	316.97	198.57	118.17	117.30	121.80	103.62	105.49	112.17	110.67	125.25
Method Recovery, %	80	122	99	88	104	93	91	95	93	98
2	343.81	199.99	118.46	118.92	121.60	104.17	106.92	112.45	112.06	127.02
Method Recovery, %	105	134	99	90	104	93	93	96	95	99
3	337.03	202.17	121.54	119.80	123.65	107.25	112.20	115.48	112.95	127.42
Method Recovery, %	99	136	102	91	106	96	98	99	95	100
Xo Avg.	332.60	200.24	119.39	118.67	122.35	105.01	108.20	113.03	111.89	126.56
k	228.48	52.76	9.76	20.23	7.24	1.71	4.90	6.34	7.98	17.78
Method Recovery, % Avg.	95	134	100	89	105	94	94	97	94	99
7 days at 4°C										
1	317.58	175.65	108.44	111.68	107.92	97.68	99.66	103.89	105.06	117.95
Preservation Recovery, %	95	87	90	94	88	93	92	91	93	93
2	315.42	191.72	111.43	120.87	116.16	104.56	105.88	110.11	109.31	126.79
Preservation Recovery, %	94	95	93	101	94	99	97	97	97	100
3	316.57	191.61	110.00	118.44	114.40	102.85	103.40	106.92	108.32	120.30
Preservation Recovery, %	95	95	92	99	93	97	95	94	96	95
Preservation Recovery, % Avg.	95	93	92	98	92	96	95	94	96	96

Note; Replicate 1, at zero time, values are from peak height measurements. Dose was doubled due to high background. The fifteen percent Et_2O fraction appears to have no Group IX in it. All values are from the 6% Et_2O fraction only.

Table A124.
ANALYSIS OF WASTEWATER 1 DOSED WITH PCB-1242

	Dose - 28 ppb								
Replicate	Peak#1	Peak#2	Peak#3	Peak#4	Peak#5	Peak#6	Peak#7	Peak#8	Peak#9
Zero Time									
1	32.37	28.36	27.58	27.58	28.00	28.00	27.72	28.00	28.81
Method Recovery, %	98	97	98	96	93	97	95	94	92
2	32.37	29.60	28.78	28.62	29.18	28.67	28.81	28.76	29.23
Method Recovery, %	98	101	102	99	98	99	99	96	93
3	33.74	29.74	28.95	28.62	29.18	28.67	28.53	28.50	28.81
Method Recovery, %	103	102	103	99	98	99	98	96	92
Xo Avg.	32.83	29.23	28.44	28.27	28.79	28.45	28.35	28.42	28.95
k	4.98	1.19	0.22	0.83	1.83	0.91	1.11	1.75	3.13
Method Recovery, % Avg.	99	100	101	98	96	98	97	95	92
7 days at 4°C									
1	30.80	27.72	26.88	26.60	26.88	27.16	26.60	27.16	28.00
Preservation Recovery, %	93	94	94	94	93	95	93	95	96
2	31.36	28.00	27.16	26.88	27.16	27.16	26.60	27.16	27.72
Preservation Recovery, %	95	95	95	95	94	95	93	95	95
3	30.80	27.16	26.32	26.60	27.44	27.16	26.60	27.16	29.12
Preservation Recovery, %	93	92	92	94	94	95	93	95	100
Preservation Recovery, % Avg.	94	94	94	94	94	95	93	95	97

Note: All values are from peak height measurements.

Table A125.
ANALYSIS OF WASTEWATER 1 DOSED WITH PCB-1248

	Dose - 40 ppb						
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
Zero Time							
1	42.28	40.76	40.44	40.16	40	40.56	40.88
Method Recovery, %	98	101	100	99	98	100	99
2	42.80	41.24	40.28	40.52	40	40.40	40.64
Method Recovery, %	99	102	99	100	98	99	98
3	42.80	41.00	40.28	40.36	40.44	40.28	40.40
Method Recovery, %	99	101	99	100	99	99	97
Xo Avg.	42.63	41.00	40.33	40.35	40.15	40.41	40.64
k	3.07	0.47	0.58	0.47	0.80	0.67	1.44
Method Recovery, % Avg.	99	101	99	100	98	99	98
7 days at 4°C							
1	44.76	42.92	42.72	42.28	41.80	42.40	42.52
Preservation Recovery, %	105	105	106	105	104	105	105
2	41.08	39.76	40.28	39.60	39.72	38.88	40.00
Preservation Recovery, %	96	97	100	98	99	96	98
3	42.32	41.04	41.28	40.72	40.60	39.76	40.60
Preservation Recovery, %	99	100	102	101	101	98	100
~~Preservation~~ Recovery, % Avg.	100	101	103	101	101	100	101

Note: All values are from peak height measurements.

Table A126.
ANALYSIS OF WASTEWATER 2 DOSED WITH GROUP I.

	Dose, ppb							
Replicate	2 alpha BHC	2 beta BHC	2 delta BHC	2 Heptachlor Epoxide	3 DDE	6 DDD	8 DDT	15 Endosulfan Sulfate
Zero time								
1	1.954	1.860	1.764	1.806	2.532	5.472	6.920	17.085
Method Recovery, %								
2	2.208	2.030	1.900	2.050	2.646	5.940	7.768	17.28
Method Recovery, %								
3	2.002	1.948	1.684	1.814	2.733	5.628	7.352	16.995
Method Recovery, %								
Xo Avg.	2.054	1.946	1.782	1.89	2.637	5.680	7.346	17.12
k								
7 days at 4°C								
1	2.22	2.168	2.054	2.100	2.793	6.192	7.752	14.61
Preservation Recovery,%	108	111	115	111	106	109	106	85
2	1.853*	2.770	2.382	2.222	2.844	6.33	8.08	14.445
Preservation Recovery,%	90	142	134	118	108	111	110	84
3	2.264	2.156	2.104	2.076	2.772	6.132	7.832	15.405
Preservation Recovery,%	110	111	118	110	105	108	107	90
Preservation Recovery % Avg.	103	121	122	113	106	109	107	87

Note: Mercury clean up was used on these dosed samples. Satisfactory results were not obtained on undosed replicates treated with mercury. Hence, without "k" values "Method Recoveries" could not be calculated.

*This is the only peak where the printout was used (peak was off chart).

Table A127.
ANALYSIS OF WASTEWATER 2 DOSED WITH GROUP II.

	Dose, ppb							
Replicate	2 gamma BHC	1 Heptachlor	2 Aldrin	3 Endosulfan I	3 Dieldrin	5 Endrin	5 Endosulfan II	12 Endrin Aldehyde
Zero Time								
1 Method Recovery, %	2.128	1.240	1.920	3.219	3.243	5.450	4.695	2.388
2 Method Recovery, %	2.092	1.250	1.902	3.159	3.168	5.435	5.445	2.088
3 Method Recovery, %	2.162	0.981	2.010	3.126	3.009	5.180	4.995	1.620
Xo Avg.	2.127	1.157	1.944	3.168	3.140	5.385	5.045	2.032
Method Recovery,% Avg.								
7 days at 4°C								
1 Preservation Recovery, %	1.962 92	0.771 67	1.408 72	3.222 102	3.258 104	5.505 102	4.720 94	4.416 217
2 Preservation Recovery, %	2.072 97	0.885 76	1.448 74	2.985 94	3.021 96	5.165 96	4.375 87	2.940 145
3 Preservation Recovery, %	2.070 97	0.655 57	1.470 76	3.084 97	3.042 97	5.060 94	3.995 79	2.280 112
Preservation Recovery % Avg.	96	67	74	98	99	97	86	158

Note: Mercury clean up was used on these dosed samples. Satisfactory results were not obtained on undosed replicates treated with mercury. Hence, without "k" values "Method Recoveries" could not be calculated.

Table A128.
ANALYSIS OF WASTEWATER 2 DOSED WITH CHLORDANE

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
	Dose = 20 ppb					
Zero Time						
1	40.604	66.622	18.531	17.70*	17.94*	17.60*
Method Recovery, %	148	0	93	89	90	88
2	56.679	98.084	27.540	20.16*	20.10*	20.22*
Method Recovery, %	228	114	138	101	101	101
3	73.344	79.656	23.189	21.16*	20.88*	20.94*
Method Recovery, %	312	22	116	106	104	105
Xo Avg.	56.875	81.454	23.086	19.673	19.64	19.586
k	11.018	75.339	0	0	0	0
Method Recovery % Avg.	229	31	115	98	98	98
7 days at 4°C						
1	103.377	86.897	24.235	19.68*	19.52*	19.64*
Preservation Recovery, %	182	107	105	100	99	100
2	93.989	88.591	38.187	19.02*	19.22*	19.02*
Preservation Recovery, %	165	109	165	97	98	97
3	97.509	91.035	44.925	19.50*	19.28*	19.24*
Preservation Recovery, %	171	112	195	99	98	98
Preservation Recovery % Avg.	172	109	155	99	98	99

Note: *These values are from peak height measurements.

Table A129.
ANALYSIS OF WASTEWATER 2 DOSED WITH TOXAPHENE

	Dose = 200 ppb							
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
Zero Time								
1	201.40	192	194.4	194.8	191.4	193.2	188.2	180.4
Method Recovery, %	101	96	97	97	96	97	94	90
2	211	202.8	204.4	204.4	203.2	202.4	199.02	195.2
Method Recovery, %	106	101	102	103	102	101	99	97
3	191.40	187	186.2	184.6	176.60	184.20	182.20	(358.2)
Method Recovery, %	96	94	93	92	88	92	91	179
Xo Avg.	201.266	193.933	195.0	194.6	190.4	193.266	189.806	187.8
k	0	0	0	0	0	0	0.165	1.097
Method Recovery % Avg.	101	97	98	97	95	97	95	93
7 days at 4°C								
1	190.40	180.20	183.40	187.20	182.20	183.00	178.20	170.00
Preservation Recovery, %	95	93	94	96	96	95	94	91
2	199.40	185.60	190.40	197.40	192.20	194.40	183.40	186.00
Preservation Recovery, %	99	96	98	202	101	101	97	99
3	211.80	197.80	201.80	206.00	189.00	203.20	195.00	(359.6)
Preservation Recovery, %	105	102	103	106	99	105	103	-
Preservation Recovery % Avg.	100	97	98	101	99	100	98	95

Note: All values are from peak height measurements. Values in parentheses were not included in calculation of averages.

Table A130.
ANALYSIS OF WASTEWATER 2 DOSED WITH PCB-1254

	Dose = 40 ppb						
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
Zero Time							
1	37.224	39.577	40.108	39.273	37.599	40.247	40.058
Method Recovery, %	93	99	100	98	94	101	105
2	39.25	42.347	43.180	41.969	39.232	42.448	32.807
Method Recovery, %	98	108	108	105	98	106	107
3	37.195	40.404	40.713	39.107	35.032	39.330	37.902
Method Recovery, %	93	101	102	98	88	98	95
Xo Avg.	37.889	40.776	41.333	40.116	37.287	40.675	40.922
k	0	0	0.077	0	0	0	0
Method Recovery % Avg.	95	102	103	100	93	102	102
7 days at 4°C							
1	43.28	37.12	36.92	34.92	35.12	35.20	33.12
Preservation Recovery, %	114	91	89	87	94	87	81
2	46.24	39.04	39.36	36.80	37.04	37.04	35.00
Preservation Recovery, %	122	96	95	92	99	91	86
3	45.68	38.52	38.08	35.76	36.20	35.96	34.72
Preservation Recovery, %	121	94	92	89	97	88	85
Preservation Recovery % Avg. 119	119	94	92	89	97	89	84

Note: Seven-day values are from peak height measurements.

Table A131.
ANALYSIS OF WASTEWATER 2 DOSED WITH PCB-1260

Dose = 80 ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10	Peak #11
Zero Time											
1	82.80	81.235	103.16	75.284	75.284	74.762	74.534	74.830	81.404	71.524	71.648
Method Recovery, %	104	98	91	94	93	93	92	92	84	89	90
2	85.12	82.056	105.553	70.171	72.130	70.24	70.398	71.746	87.345	66.950	69.20
Method Recovery, %	106	99	94	87	89	88	87	89	92	84	87
3	93.68	94.327	109.824	82.734	86.718	80.40	82.02	79.511	90.235	77.628	75.36
	117	114	100	103	108	101	102	98	95	97	94
Xo Avg.	87.20	85.872	106.179	76.063	77.876	75.13	75.65	75.362	86.328	72.034	72.069
k	0	3.048	30.154	0.237	0.622	0	0.691	0.939	14.044	0	0
Method Avg. % Recovery	109	104	95	95	97	94	94	93	90	90	90
7 days at 4°C											
1	80.733	103.463	109.347	76.838	78.929	75.52	78.60	72.517	96.763	74.173	73.60
% Recovery	93	120	103	101	101	101	014	96	112	103	102
2	79.36	87.412	99.239	62.699	61.778	61.84	63.735	60.972	82.036	60.56	61.12
% Recovery	91	102	93	82	79	82	84	81	95	84	85
3	78.80	98.443	112.039	79.393	79.192	77.76	77.892	76.148	98.361	76.40	75.04
% Recovery	90	115	106	104	102	104	103	101	114	106	104
Preservation Avg. % Recovery	91	112	101	96	94	95	97	93	107	98	97

Note: All values obtained by peak height measurements. The fifteen percent Et_2O fraction appears to have no Group VII in it. All values are from the 6% Et_2O fraction only.

Table A132.
ANALYSIS OF WASTEWATER 2 DOSED WITH PCB-1242

Replicate	Dose = 28 ppb								
	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9
Zero Time									
1	37.744	28.476	44.32	30.856	32.90	40.18	29.372	27.272	27.496
Method Recovery, %									
2	38.612	27.44	45.584	35.98	32.62	36.848	27.72	25.48	26.152
Method Recovery, %									
3	41.076	29.176	47.012	35.00	34.44	33.348	28.504	27.188	28.00
Method Recovery, %									
Xo Avg.	39.144	28.364	45.638	33.945	33.32	36.792	28.532	26.646	27.216
k									
Method Avg. % Recovery									
7 days at 4°C									
1	40.684	28.868	45.472	29.988	34.636	37.044	29.96	27.608	28.588
Preservation, % Recovery	104	102	100	88	104	101	105	104	105
2	42.42	25.844	51.268	31.528	37.184	40.74	48.888	32.228	30.744
Preservation, % Recovery	108	91	112	93	112	111	171	121	113
3	39.984	30.464	45.332	30.856	34.804	30.968	30.856	29.764	43.764
Preservation, % Recovery	102	107	99	91	104	84	108	112	161
Preservation Avg. % Recovery	105	100	104	91	107	99	128	112	126

Note: see note on Table A126. All values obtained by peak height measurements.

Table A133.
ANALYSIS OF WASTEWATER 2 DOSED WITH PCB-1248

	Dose = 40 ppb						
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
Zero Time							
1 Method Recovery, %	41.88	43.157	40.300	41.760	42.392	45.883	41.297
2 Method Recovery, %	40.423	40.008	38.555	39.277	39.924	43.185	38.661
3 Method Recovery, %	39.931	42.002	39.414	38.412	39.734	42.501	38.655
Xo Avg.	40.744	41.722	39.423	39.816	40.683	43.856	39.537
k							
Method Recovery, Avg. %							
7 days at 4°C							
1 Preservation, % Recovery	36.932 91	65.494 157	35.048 89	37.487 94	37.860 93	39.103 89	36.555 92
2 Preservation, % Recovery	36.919 91	68.693 165	36.041 91	36.502 92	37.249 92	38.335 87	36.043 92
3 Preservation, % Recovery	sample spilled as vial broke						
Preservation Avg. % Recovery	91	161	90	93	92	88	92

Note: see note on Table A126

Table A134.
ANALYSIS OF WASTEWATER 3 DOSED WITH GROUP I

	Dose, ppb							
	1	2	2	2	3	6	8	15
Replicate	alpha BHC	beta BHC	delta BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
Zero time								
1	.998	1.834	1.626	1.859	2.555	5.373	7.175	15.035
Method % Recovery	84	81	75	91	84	90	90	99
2	.976	1.829	1.761	1.892	2.591	5.515	6.970	15.081
Method % Recovery	82	81	81	93	86	92	87	99
3	.933	1.760	1.740	1.820	2.502	5.246	6.696	16.539
Method % Recovery	78	77	80	89	83	87	84	109
Xo Avg.	.969	1.807	1.709	1.857	2.549	5.378	6.947	15.551
"k" background, Avg.	.154	.213	.134	.038	.022	0	0	.181
Method Avg. % Recovery	82	80	79	91	84	90	87	102
7 days at 4°C								
1	.998	1.797	1.723	1.876	2.175	4.770	5.816	13.847
Preservation % Recovery	103	99	101	101	85	89	84	89
2	1.014	1.826	1.701	1.920	2.262	4.945	6.086	14.871
Preservation % Recovery	105	101	100	103	89	92	88	96
3	0.994	1.800	1.702	1.886	2.239	4.898	6.073	14.273
Preservation % Recovery	103	100	100	102	88	91	87	92
Preservation Avg. % Recovery	103	100	100	102	87	91	86	92

Note: Mercury cleanup used.

Table A135.
ANALYSIS OF WASTEWATER 3 DOSED WITH GROUP II

	Dose, ppb							
	1	1	2	3	3	5	5	12
Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
Zero time								
1 Method Recovery, %	1.014 96	.913 89	1.927 93	2.940 94	2.815 93	4.678 94	4.972 83	10.059 84
2 Method Recovery, %	.993 94	.881 85	1.910 92	2.976 96	2.846 94	4.789 96	4.933 82	10.621 89
3 Method Recovery, %	1.035 98	.929 90	1.989 96	2.969 95	2.857 94	4.790 96	5.146 86	10.143 85
Xo Avg.	1.014	.907	1.942	2.961	2.839	4.752	5.017	10.274
"k" background, Avg.	.052	.028	.072	.106	.038	0	.847	0
Method Avg. % Recovery	96	88	94	95	94	95	84	86
7 days at 4°C								
1 Preservation Recovery, %	1.047 103	0.799 88	1.603 83	2.915 98	2.764 97	4.553 96	4.969 99	9.344 91
2 Preservation Recovery, %	1.110 109	0.817 90	1.630 84	3.008 102	2.890 102	4.771 100	5.139 102	9.630 94
3 Preservation Recovery,%	1.074 106	0.817 90	1.584 82	2.897 98	2.826 100	4.713 99	5.169 103	9.537 93
Preservation Avg. % Recovery	106	89	83	99	100	98	101	92

Note: Mercury cleanup used. At zero time, gamma-BHC and endrin aldehyde values are from peak height measurements.

Table A136.
ANALYSIS OF WASTEWATER 3 DOSED WITH CHLORDANE

Replicate	Dose = 20 ppb Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
Zero time						
1 Method % Recovery	19.500 87	15.880 78	17.760 85	18.840 91	18.840 91	18.320 92
2 Method % Recovery	19.500 87	16.440 81	17.760 85	18.620 90	18.660 90	16.660 83
3 Method % Recovery	18.580 93	15.480 76	17.660 85	17.820 86	17.860 86	17.820 89
xo avg	19.283	15.933	17.726	18.426	18.453	17.600
"k" background, avg.	1.048	.305	.698	.562	.593	0
Method Avg. % Recovery	86	78	85	89	89	88
7 days at 4°C						
1 Preservation % Recovery	18.96 98	16.18 102	18.18 103	17.6 96	18.06 98	17.44 99
2 Preservation % Recovery	19.82 103	16.96 106	19.00 107	18.30 99	18.70 101	18.18 103
3 Preservation % Recovery	19.36 100	16.46 103	18.84 106	18.48 100	18.96 103	18.18 103
Preservation Avg. % Recovery	101	104	105	98	101	102

Note: Mercury cleanup used. All values at zero time are from peak height measurements.

Table A137.

ANALYSIS OF WASTEWATER 3 DOSED WITH TOXAPHENE

	Dose = 200 ppb							
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8
Zero time								
1	187.0	176.8	176.6	185.8	172.4	171.6	162.6	150.0
Method % Recovery	93	88	88	93	86	86	81	75
2	202.2	194.0	194.2	201.0	191.4	194.0	188.4	184.8
Method % Recovery	101	97	97	101	96	97	94	92
3	203.5	193.2	192.8	199.0	189.0	190.4	183.8	174.6
Method % Recovery	102	97	96	100	95	95	92	87
Xo Avg.	197.57	188	187.9	195.3	184.3	185.3	178.3	169.8
"k" background, Avg.	0.400	0	0	0	0	0	0.281	0
Method Avg. % Recovery	99	94	94	98	92	93	89	85
7 days at 4°C								
1	169.2	157.6	158.9	167	151.8	158.2	148.4	137.4
Preservation % Recov.	86	84	85	86	82	85	83	81
2	173	161	163.6	171.4	157.2	163.6	155.6	144.6
Preservation % Recov.	88	86	87	88	85	88	87	85
3	172.8	159.4	171.4	170.4	168.4	161	151.6	140
Preservation % Recov.	87	85	91	87	91	87	85	82
Preservation Avg. % Recovery	86	85	88	87	86	87	85	83

Note: Mercury cleanup used. All values are from peak height measurements.

Table A138
ANALYSIS OF WASTEWATER 3 DOSED WITH PCB-1254

	Dose =40 ppb						
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
Zero time							
1	37.88	38.04	36.44	36.24	36.52	36.48	35.88
Method % Recovery	93	94	89	90	91	91	90
2	37.64	37.96	36.20	35.84	35.28	36.12	35.44
Method % Recovery	93	94	89	89	88	90	89
3	37.84	38.16	36.44	36.24	37.00	36.44	35.88
Method % Recovery	93	94	89	90	93	91	90
Xo Avg.	37.79	38.05	36.36	36.11	36.27	36.35	35.73
"k" background	0.514	0.441	0.641	0.381	0	0	0
Method AVg. % Recovery	93	94	89	89	91	91	89
7 days at 4°C							
1	35.00	35.24	33.36	32.92	30.16	33.00	32.12
Preservation % Recov.	93	93	92	91	83	91	90
2	35.60	35.64	34.12	34.00	33.20	33.52	32.44
Preservation % Recov.	94	94	94	94	92	92	91
3	36.72	37.20	35.48	35.20	35.20	35.56	34.16
Preservation % Recov.	97	98	98	97	97	98	96
Preservation Avg. % Recovery	95	95	95	94	91	94	92

Note: Mercury cleanup used. All values are from peak height measurement.

Table A139
ANALYSIS OF WASTEWATER 3 DOSED WITH PCB-1016

	Dose = 25 ppb							
Replicate	**Peak #1**	**Peak #2**	**Peak #3**	**Peak #4**	**Peak #5**	**Peak #6**	**Peak #7**	**Peak #8**
Zero Time								
1	24.200	23.500	22.975	23.125	23.550	22.575	22.750	23.800
Method % Recovery	97	94	92 (90)	93	94 (76)	90	91	95 (57)
2	25.425	24.575	24.025	24.675	25.000	23.675	23.750	24.025
Method % Recovery	102	98	96 (95)	99	100 (81)	95	95	96 (58)
3	24.750	23.350	23.900	23.300	24.100	23.075	22.975	24.750
Method % Recovery	99	93	96 (94)	93	96 (78)	92	92	99 (61)
Xo Avg.	24.791	23.808	23.633	23.700	24.216	23.108	23.158	24.191
"k" background	35.150	3.662	.370	0	4.674	1.120	.221	9.470
Method Avg. % Recov	99	95	95 (93)	95	97 (78)	92	93	97 (59)
7 days at 4°C								
1	24.575	22.525	22.900	21.900	22.300	21.125	21.550	22.500
Preservation % Recov.	99	95	97	92	92	91	93	93
2	24.275	22.500	22.600	21.950	21.975	20.850	21.200	22.500
Preservation % Recov.	98	95	96	93	91	90	92	93
3	23.800	22.375	22.575	21.875	22.550	20.850	21.050	22.500
Preservation % Recov.	96	94	96	92	93	90	91	93
Preservation Avg. % Recovery	98	95	96	92	92	90	92	93

Note: Mercury cleanup used. All values are from peak height measurements. Where the background peak retention times disagree with the retention times of peaks from the standard, the "k" has not been subtracted. Where agreement in retention times occur, the recoveries have been calculated both ways, i.e. not subtracting and subtracting the "k" value. The values in ( ) are with "k" subtracted. The effect of Hg cleanup is variable. It appears that in these three instances, peaks 3, 5, 8, the background peaks were completely removed from the spiked samples. In a trial run earlier, with one sample, the peak reductions were only partial so the "k" values were obtained without Hg cleanup.

Table A140
ANALYSIS OF WASTEWATER 3 DOSED WITH PCB-1260

	Dose ≈80 ppb										
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10	Peak #11
Zero Time											
1	76.800	76.706	72.965	72.634	72.265	72.087	72.335	70.947	70.270	70.955	71.232
Method % Recovery	96	96	91	91	90	90	90	89	84	89	89
2	76.800	78.152	74.675	74.274	73.612	73.536	73.635	72.537	73.414	73.197	73.425
Method % Recovery	96	98	93	93	92	92	92	91	88	91	92
3	75.040	76.812	72.379	72.367	72.120	71.844	72.076	71.439	71.434	70.869	71.116
Method % Recovery	94	96	90	90	90	90	90	89	86	89	89
Xo Avg.	76.216	77.223	73.339	73.091	72.665	72.489	72.682	71.641	71.706	71.673	71.924
"k" background	0	0	0	0	0	0	0	0	3.018	0	0
Method Avg. % Recov.	95	97	91	91	91	91	91	90	86	90	90
7 days at 4°C											
1	71.360	72.505	67.742	67.614	67.784	66.805	67.370	66.401	66.098	66.110	65.982
Preservation % Recov.	94	94	92	93	93	92	92	93	92	92	92
2	70.000	70.873	65.984	66.140	66.246	65.244	64.696	65.845	64.530	65.072	67.200
Preservation % Recov.	92	92	90	90	91	90	89	92	90	91	93
3	71.360	71.745	66.779	66.801	66.905	66.161	66.666	65.495	65.305	65.805	66.758
Preservation % Recov.	94	93	91	91	92	91	92	91	91	92	93
Preservation Avg. % Recovery	93	93	91	91	92	91	91	92	91	92	93

Note: Mercury cleanup used. Values for Peak 1 are from peak height measurements. The fifteen percent Et_2O fraction appears to have no Group VII in it. All values are from the 6% Et_2O fraction only.

Table A141
ANALYSIS OF WASTEWATER 3 DOSED WITH PCB-1221

Replicate	Dose = 110 ppb						
	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
Zero Time							
1	105.93	108.57	108.24	(59.557) 151.69	105.27	105.60	105.71
Method % Recovery	96	99	98	138 (54)	96	96	93
2	105.27	108.13	108.46	(70.997) 163.13	105.16	105.93	105.27
Method % Recovery	96	98	99	148(65)	96	96	93
3	110.22	113.63	114.73	(76.497) 168.63	110.44	111.10	112.09
Method % Recovery	100	103	104	153(70)	100	101	99
Xo Avg.	107.140	110.110	110.476	161.150	106.956	107.543	107.690
"k" background	0	0	0	92.133	0	0	3.261
Method Avg. % Recov	97	100	100	146 (63)	97	98	95
7 days at 4°C							
1	84.920	91.630	95.480	131.340	92.510	92.840	91.080
Preservation % Recov.	79	83	86	82	87	86	85
2	97.680	101.420	101.530	142.780	97.240	94.710	63.500
Preservation % Recov.	91	92	92	89	91	88	87
3	107.030	110.000	108.680	154.110	105.050	103.290	100.540
Preservation % Recov.	100	100	98	96	98	96	93
Preservation Avg. % Recovery	90	92	92	89	92	90	88

Note: Mercury cleanup used. All values are from peak height measurements.
See note on Table 149.

Table A142.
ANALYSIS OF WASTEWATER 3 DOSED WITH PCB-1232

	Dose =110 ppb									
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak #8	Peak #9	Peak #10
Zero Time										
1	109.23	180.07 (29.263)	94 71	90.53	87.56 (81.879)	90.20	92.73 (52.460)	86.24	87.45 (76.342)	94.16 (64.647)
Method % Recovery	99	164 (27)	86	82	80 (74)	82	84 (49)	78	80 (69)	86 (59)
2	112.69	195.5 (45.064)	100.79	97.07	92.87 (87.189)	95.72	98.11 (55.450)	92.18	90.50 (79.592)	97.87 (68.357)
Method % Recovery	102	178 (41)	92	88	84 (79)	87	89 (50)	84	82 (72)	89 (62)
3	106.39	189.05 (38.614)	97.91	94.64	90.83 (85.149)	93.33	95.64 (55.370)	88.80	88.8 (77.692)	96.36 (66.847)
Method % Recovery	97	172 (35)	89	86	83 (77)	85	87 (50)	81	81 (71)	88 (61)
Xo Avg.	109.44	188.21	97.80	94.08	90.42	93.08	95.49	89.07	88.92	96.13
"k" background, Avg.	0	150.436	0	0	5.681	0	40.270	0	11.108	29.513
Method Avg. % Recov.	99	171 (34)	89	85	82 (77)	85	87 (49)	81	81 (71)	88 (61)
7 days at 4°C										
1	102.52	160.38	95.04	85.80	83.49	82.61	83.27	75.46	77.55	80.08
Preservation % Recov.	94	107	97	91	92	89	87	85	87	83
2	98.67	157.63	93.61	85.80	83.49	83.49	83.82	76.23	77.11	81.62
Preservation % Recov.	90	105	96	91	92	90	88	86	87	85
3	100.98	168.30	96.80	89.21	86.79	86.57	88.55	80.41	80.41	84.59
Preservation % Recov.	92	112	99	95	96	93	93	90	90	88
Preservation Avg. % Recovery	92	108	97	92	93	91	89	87	88	85

Note: Mercury cleanup used. All values are from peak height measurements. See note on Table A139. The fifteen percent Et_2O fraction appears to have no Group IX in it. All values are from the 6% Et_2O fraction only.

Table A143

ANALYSIS OF WASTEWATER 3 DOSED WITH PCB-1242

Dose = 56ppb

Replicate	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8	Peak 9
Zero Time									
1	49.11	46.20	44.74(44.36)	46.03	47.04(40.872)	43.57	44.02	43.96	46.59(44.84)
Method % Recovery	83	83	80(79)	82	84(73)	78	79	79	83(80)
2	50.40	46.87	45.86(45.48)	46.65	48.27(42.102)	43.96	44.46	44.18	46.93(45.18)
Method % Recovery	90	84	82(81)	83	86(75)	79	79	79	84(81)
3	49.55	46.65	46.20(45.82)	47.38	48.27(42.102)	45.42	45.92	46.20	48.72(46.97)
Method % Recovery	88	83	83(82)	85	86(75)	81	82	83	87(84)
Xo Avg.	49.686	46.573	45.600	46.686	47.860	44.316	44.800	44.780	47.413
"k" background	0	0	.380	0	6.168	0	0	0	1.750
Method Avg. % Recov	87	83	82(81)	83	85(74)	79	80	80	85(82)
7 days at 4°C									
1	54.77	52.25	50.85	51.18	51.35	48.38	48.33	48.72	51.41
Preservation % Recovery	110	112	112	110	107	109	108	109	108
2	55.55	52.70	51.52	51.80	52.25	49.06	49.62	49.17	52.14
Preservation % Recovery	112	113	113	111	109	111	111	110	110
3	46.00	53.59	52.75	53.48	53.54	50.74	50.90	51.24	54.99
Preservation % Recovery	113	115	116	115	112	114	114	114	116
Preservation Avg. % Recovery	112	113	114	112	109	111	111	111	111

NOTE: Mercury cleanup used. All values are from peak height measurements. See note on Table A139.

Table A144
ANALYSIS OF WASTEWATER 3 DOSED WITH PCB-1248

				Dose = 40ppb			
Replicate	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7
Zero Time							
1	35.80	34.84(33.87)	33.20	33.80	37.00	33.60(33.19)	34.64
Method % Recovery	90	87(85)	83	85	93	84(83)	87
2	35.68	34.68(33.71)	32.72	33.68	35.24	32.64(32.23)	35.52
Method % Recovery	89	87(84)	82	84	88	82(81)	89
3	35.60	34.60(33.63)	33.40	33.88	36.44	33.76(33.35)	35.00
Method % Recovery	89	87(84)	84	85	91	84(83)	88
Xo Avg.	35.69	34.71	33.11	33.79	36.23	33.33	35.05
"k" background	0	.972	0	0	0	.413	0
Method Avg. % Recov	89	87(84)	83	85	91	83	88
7 days at 4°C							
1	34.66	33.23	30.76	31.43	32.82	30.75	31.93
Preservation % Recov.	97	96	93	93	91	92	91
2	33.82	33.33	30.99	31.66	33.09	30.97	31.93
Preservation % Recov.	95	96	94	94	91	93	91
3	34.29	33.38	31.09	31.78	32.90	30.90	31.48
Preservation % Recov.	96	96	94	94	91	93	90
Preservation Avg. % Recovery	96	96	93	94	91	93	91

NOTE: Mercury cleanup used. All values are from peak height measurements. See note on Table A139.

Table A145.
ANALYSIS OF WASTEWATER 4 DOSED WITH GROUP I

	Dose, ppb							
	1	2	2	2	3	6	8	15
Replicate	alpha BHC	beta BHC	delta BHC	Heptachlor Epoxide	DDE	DDD	DDT	Endosulfan Sulfate
1	1.032	2.216	1.750	2.038	2.885	5.782	7.326	16.217
Method Recovery,%	82	98	85	98	95	96	91	101
2	1.028	2.188	1.740	2.041	2.841	5.704	7.379	16.055
Method Recovery,%	82	97	85	98	94	94	92	100
3	1.017	2.170	1.841	2.042	2.865	5.731	7.646	16.670
Method Recovery,%	81	96	90	98	95	95	95	104
Xo Avg.	1.026	2.191	1.777	2.040	2.857	5.739	7.450	16.314
k*	.211	.250	.050	.088	.030	.035	.024	1.122
Method Recovery % Avg.	82	97	86	98	94	95	93	101

*k calculated from undosed replicates 5 and 6.

Table A146.
ANALYSIS OF WASTEWATER 4 DOSED WITH CHLORDANE

Dose = 20ppb

Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
1	20.800	16.677	19.417	19.952	19.888	18.540
Method Recovery,%	96	76	91	94	93	89
2	21.470	17.297	20.126	20.672	20.503	19.568
Method Recovery,%	100	79	95	98	96	94
3	20.685	16.734	19.119	19.716	19.626	18.000
Method Recovery,%	96	76	89	93	91	86
Xo Avg.	20.985	16.903	19.554	20.113	20.006	18.703
k*	1.534	1.567	1.225	1.142	1.332	0.799
Method Recovery % Avg.	97	77	92	95	93	90

Note: All values are from peak height measurements.

* k calculated from undosed replicates 5 and 6.

Table A147.
ANALYSIS OF WASTEWATER 4 DOSED WITH PCB-1254

				Dose = 40ppb			
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1	40.110	42.260	38.255	36.765	37.671	36.681	36.810
Mehtod Recovery,%	99	101	95	91	94	92	92
2	40.00	41.948	41.848	37.526	39.024	37.608	37.456
Method Recovery,%	99	101	104	93	98	94	94
3	40.690	42.772	42.771	38.130	38.537	37.938	37.515
Method Recovery,%	100	103	106	95	96	95	94
Xo Avg.	40.267	42.327	40.961	37.474	38.411	37.409	37.260
k*	0.537	1.748	0.407	0.186	0	0	0
Method Recovery % Avg.	99	101	101	93	96	94	93

Note: All values are from peak height measurements.

* k calculated from undosed replicates 5 and 6.

TABLE A148.
ANALYSIS OF WASTEWATER 5 DOSED WITH GROUP II.

	Dose,ppb							
	1	1	2	3	3	5	5	12
Replicate	gamma BHC	Heptachlor	Aldrin	Endosulfan I	Dieldrin	Endrin	Endosulfan II	Endrin Aldehyde
1	1.306	3.543	2.204	7.953	66.190	31.608	7.473	101.097
Method Recovery,%	107	57	94	101	303	100	92	97
2	1.265	3.570	2.146	8.005	67.279	30.614	7.195	104.881
Method Recovery,%	103	60	91	103	340	80	86	129
3	1.343	3.676	2.217	7.673	65.176	31.315	7.383	97.619
Method Recovery,%	111	70	95	92	270	94	90	68
Xo Avg.	1.305	3.596	2.189	7.877	66.220	31.179	7.350	101.199
K	.235	2.973	.327	4.918	57.089	26.609	2.891	89.402
Method Recovery,% Avg.	107	62	93	99	307	91	89	98

Note: Endrin was determined isothermally at 200°C. Other compounds were determined with the temperature programmed from 160° to 200°C at 4° per minute.

Table A149.
ANALYSIS OF WASTEWATER 5 DOSED WITH CHLORDANE

	Dose - 20 ppb					
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6
1	26.763	56.568	22.903	35.991	29.365	21.176
Method Recovery,%	101	60	110	98	94	93
2	26.667	55.154	22.857	35.031	29.245	22.353
Method Recovery,%	100	53	109	93	93	99
3	26.194	56.173	22.791	35.476	29.312	*
Method Recovery,%	98	58	109	95	93	
Xo Avg.	26.541	55.965	22.840	35.499	29.307	21.765
k	6.616	44.557	.974	16.446	10.646	2.528
Method Recovery % Avg.	100	57	109	95	93	96

Note: *Data point missed due to problem with recorder.
All values are from peak height measurements.

ANALYSIS OF WASTEWATER 5 DOSED WITH PCB-1242 + PCB-1254

	Dose = 28 ppb AR 1242							Dose = 40 ppb AR 1254						
Replicate	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7	Peak # 8 + #1	Peak #9 + #2	Peak #3	Peak #4	Peak #5	Peak #6	Peak #7
1		114.683	68.522	45.447		34.805	35.116	71.259	66.006	38.368	37.237	37.438	37.860	36.979
Method Recovery, %		41	68	68		100	66	84	89	85	88	86	88	88
2		106.34	64.021	41.277		28.354	31.234	64.958	61.914	35.714	35.495	35.847	35.062	35.408
Method Recovery, %		11	52	53		77	52	75	83	78	83	85	81	84
3		106.75	63.340	39.490		28.00	31.974	64.828	61.284	35.102	35.195	35.961	34.897	35.045
Method Recovery, %		13	49	47		75	55	75	82	77	82	83	80	83
Xo Avg.		109.19	65.294	42.071		30.386	32.775	67.015	62.105	36.395	35.976	36.749	35.940	35.811
k		103.25	49.490	26.383		6.911	16.634	13.904	5.247	4.327	2.222	2.956	2.798	1.813
Method Recovery % Avg.		22	56	56		84	58	78	84	80	84	84	83	85